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# ANALYTICAL ABSTRACTS

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# ANALYTICAL ABSTRACTS

## 1.—GENERAL ANALYTICAL CHEMISTRY

**3519. Improved Dumas method for molecular-weight determination.** H. H. Anderson and L. D. Shubin (Drexel Inst. of Technol., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 852–854.—The modification of Dumas bulbs is described. When unsealed, these bulbs can be used repeatedly for liquids boiling between 105° and 200°. They all have proportions similar to those of the one illustrated for acetic acid—a 4·511-ml bulb, and an upper neck tapering to 1·0 mm int. diam. Details are given of a titration technique for the determination of the available acidity of such compounds as triethylgermanium bromide; this technique is accurate enough to replace weighing.
E. G. CUMMINS

**3520. Use of anthranilic acid in chemical analysis.** K. B. Yatsimirskiĭ and V. V. Kharitonov. *Trudȳ Ivanovsk. Khim.-Tekhnol. Inst.*, 1956, (5), 6–15; *Ref. Zhur., Khim.*, 1957, Abstr. No. 8366.—The ions $Mn^{2+}$, $Co^{2+}$, $Ni^{2+}$, $Cu^{2+}$, $Zn^{2+}$ and $Cd^{2+}$ are not quant. pptd. by anthranilic acid (**I**) from acetate buffer soln. with high pH values unless excess of **I** is present, nor does **I** precipitate these ions from ammoniacal soln. The incomplete pptn. is connected with the formation of complexes with acetate ion and $NH_3$. In the determination of these elements with **I**, pptn. must be carried out from soln. in which complex formation is negligible and in the presence of excess of **I**. When using **I** as an analytical reagent it is necessary to add a neutral salt ($NaNO_3$) to give the soln. a high ionic strength; this increases the sensitivity of the pptn. The following solubility products have been calculated from solubility determinations in aq. $NH_3$ and acetate buffer soln.—$MnR_2$, $8·0 \times 10^{-10}$; $CoR_2$, $1·2 \times 10^{-11}$; $NiR_2$, $5·4 \times 10^{-13}$; $CuR_2$, $1·3 \times 10^{-13}$; $ZnR_2$, $3·1 \times 10^{-11}$; and $CdR_2$, $1·4 \times 10^{-10}$; $R = C_6H_4NH_2COO-$.
C. D. KOPKIN

**3521. Oxidation of the analytical reagent tiron (disodium 4:5-dihydroxybenzene-1:3-disulphonate).** G. F. Atkinson and W. A. E. McBryde (The Univ., Toronto, Ontario, Canada). *Canad. J. Chem.*, 1957, **35** (5), 477–487.—The rapid yellowing, especially at pH values >9, of alkaline soln. of tiron used in analysis is ascribed to the oxidative formation of the corresponding quinone. In certain conditions, *viz*, anodic electrolysis of 0·01 $M$ soln. at pH 10 to 12 with a very small current, oxidation with $KMnO_4$ or 30% $H_2O_2$, and titration at pH 10 with 0·1 $N$ $K_3Fe(CN)_6$, a green coloration (probably due to the semi-quinone) having an absorption max. at 650 m$\mu$ is produced. Overlap of these oxidation products and the ferric derivative will increase the light absorption at 480 m$\mu$ in the colorimetric determination of $Fe^{3+}$ by tiron at pH 8·5 (*cf.* Yoe and Jones, *Brit. Abstr. C*, 1944, 111); $Fe^{2+}$ also interfere, even in the complete

absence of O, owing to the formation of ferric tironate, $FeR_3^{9-}$. The influence of the oxidation of tiron on wave heights during the polarographic determination of Fe in $M$ NaOH (Doležal and Adam, *Chem. Listy*, 1953, **47**, 1467) is discussed. Tiron in alkaline soln. is suitable only for the determination of total Fe; a separate determination of $Fe^{2+}$ with the same reagent should be made in more acid soln.
W. J. BAKER

**3522. A new luminescent acid - base indicator.** L. P. Zharova and V. L. Zolotavin. *Trudȳ Ural'sk. Politekh. Inst.*, 1956, No. 57, 76–78; *Ref. Zhur., Khim.*, 1957, Abstr. No. 19,466.—An alcoholic soln. of 3-amino-2-chloro-5-(2-diethyl-amino-1-methylbutylamino)-7-methoxyacridine hydrochloride (**I**) gives a clear green fluorescence in u.v. light; addition of **I** to an acid soln. gives an orange fluorescence which, on making the soln. alkaline, changes to green over the pH range 6 to 8.
C. D. KOPKIN

**3523. Indicator for the carbon dioxide saturation of caustic potash.** E. Winter (BundesforschAnstalt für Lebensmittelfrischhaltung, Karlsruhe, Germany). *Z. anal. Chem.*, 1957, **156** (4), 281–283.—2-*p*-Nitrophenylazo-1-naphthol-3 : 8-disulphonic acid, disodium salt (Epsilon blue), is proposed as a saturation indicator for the absorption of $CO_2$ in KOH. Between pH 12·5 and 12·0 (*i.e.*, within the range where all available KOH is converted into $K_2CO_3$) a clearly detectable colour change from blue to red takes place.
D. F. PHILLIPS

**3524. New group of complexometric indicators.** I. S. Mustafin and E. A. Kashkovskaya (N.G. Chernȳshevskiĭ Saratov Univ.). *Zavod. Lab.*, 1957, **23** (5), 519–522.—The use of phenolcarboxylic acids of the triphenylmethane series, Chromoxan blue BLD and B, Chromoxan violet B and 5B, Chromoxan dark-blue R and Chromoxan green GG ('chrome-green'), as indicators in the EDTA titration of cations is studied. Reaction in alkaline medium occurs with Mg, Ca, Ni, Co, Mn, Cd, $Ce^{3+}$, Zn and $Cu^{2+}$, in acid medium with $Ti^{4+}$, Th, Al, $UO_2^{2+}$, $Cr^{3+}$, $V^{4+}$ and $Fe^{3+}$, and in neutral medium with Be. The colours of the dyestuff soln. over various pH ranges and the colours obtained with the cations are described, and methods of titrating $Ca^{2+}$ and $Mg^{2+}$ are outlined.
G. S. SMITH

**3525. Metallochromic indicators. I. Introduction.** J. Körbl and R. Přibil (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (2), 302–310.—Metallochromic indicators [murexide, Eriochrome black T, catechol violet, pyrogallol red, bromopyrogallol red, 3:4-dihydroxy-4'-nitroazobenzene, 3:4-dihydroxyazo-benzene-4'-sulphonic acid, phthalein complexone, Xylenol orange, methyl thymol blue and 1-(2-pyridylazo)-2-naphthol] and their mode of action are discussed.
J. ZÝKA

**3526. Use of biguanide sulphate as a reagent.**
Asit Kumar Ray (Dept. of Inorg. Chem., Indian Ass. for Cultivation of Sci., Calcutta). *Z. anal. Chem.*, 1957, **156** (1), 18–22.—Biguanide acid sulphate is suggested as a volumetric primary standard acid. Conductimetric standardisation of 0·1 N NaOH led to a value 0·8% higher than that obtained similarly with standardised $H_2SO_4$; thermometric titration of N NaOH gave a result 0·25% less with biguanide sulphate than with $H_2SO_4$. In a comparison with five standard acids, the standardisation of 0·1 N NaOH with biguanide sulphate gave a result 0·3% higher than the mean of the results with the five other acids.                    M. F. C. LADD

**3527. Complexometric titrations using azoxine indicators.** J. S. Fritz, W. J. Lane and A. S. Bystroff (Inst. for Atomic Res., Iowa State Coll., Ames, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 821–825.—Details of the prep. of some 7-arylazo-8-hydroxy-quinoline-5-sulphonic acids (azoxines) are given. These reagents form yellow complexes with $Cu^{2+}$ and $Zn^{2+}$ and other metal ions, but the 7-(1-naphthylazo) compound is chosen for detailed investigation because of the greater colour contrast between it and its metal complexes. Buffered soln. of $Cd^{2+}$, $Co^{2+}$, $Cu^{2+}$, $Pb^{2+}$, $Ni^{2+}$, $Y^{3+}$, lanthanides and $Zn^{2+}$ at pH 5·5 to 6·5, or 6·0 to 6·5 in the presence of citrate, of $Mn^{2+}$ at pH 6·5 to 7·0, and of $Fe^{3+}$ and $Th^{4+}$ at pH 3·0 to 3·5 are titrated with 0·05 M EDTA (disodium salt) with the selected azoxine as indicator. Sharp end-points are given by $Zn^{2+}$, $Cu^{2+}$, $Co^{2+}$ and $Pb^{2+}$, but with the other listed ions the addition of a small known quantity of $Cu^{2+}$ near the end-point is recommended, when the colour of the $Cu^{2+}$ - indicator complex disappears only after all the $Cu^{2+}$ and other cation has been titrated. Precision and accuracy data and the study of interferences of any single cation are discussed.                    D. A. PANTONY

**3528. Complexometric titrations. (Chelatometry.) XXVII. Naphthol violet, a new simple metal indicator.** B. Buděšínský (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (4), 726–729.—Naphthol violet [4-*p*-nitrophenylazo-2-bis(carboxymethyl)aminomethyl-1-naphthol] (I) reacts with many metal cations in acid and alkaline soln., changing its colour. The reactions with bivalent cations in alkaline soln. (Zn, Cd, Mg, Mn, Co, Cu) and with Bi in acid soln. are suitable for visual indication in the complexometric titration of those elements. *Procedure for Mg, Zn, Cd, Co, Cu and Mn*—Dissolve a few milligrams of I in aq. $NH_3$ (25%) (5 ml), add a neutral soln. of the metal ion (0·01 to 0·3 millimole in 5 to 30 ml) and titrate with 0·05 M EDTA (disodium salt). The colour change is red - violet to blue. For the titration of Co, a medium of 10% aq. $NH_3$ at 40° to 50° is recommended. In the titration of Mn, hydroxylamine must be added to prevent oxidation. A large excess of ammonium salts interferes with the colour change. Bismuth can be titrated in 0·1 M $HNO_3$; the indicator must first be dissolved in NaOH soln.                    J. ZÝKA

**3529. Standardisation of potassium permanganate with oxalic acid. I. Titration in phosphoric acid solution.** Kisaburo Tabuchi, Makoto Otomo and Hiroyuki Sakaki (Fac. of Liberal Arts, Hirosaki Univ., Hirosaki, Aomori Prefecture). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (1), 1–3.—The reduction of $KMnO_4$ with oxalic acid proceeds rapidly in 0·3 to 5 M $H_3PO_4$ at 70° to 90°. The

product appears to consist of $KH_2PO_4$ and $Mn(H_2PO_4)_2$. The error is $< \pm 0.2\%$.

**II. Titration in perchloric acid.** Kisaburo Tabuchi, Makoto Otomo and Minoru Takaoka. *Ibid.*, 1957, **78** (1), 3–5.—The oxidation of oxalic acid with $HClO_4$ proceeds only slowly at $< 100°$. When the titration is carried out in 0·5 to 3·5 N $HClO_4$ at 50° to 85°, the error is $< 0.2\%$. In the presence of $> 10$ μg of Mn per ml of the initial oxalic acid soln. in 1 to 4 N $HClO_4$, the titration can be carried out rapidly at 25°. Anions in the soln. have little effect.                    K. SAITO

**3530. Chemical analysis with the aid of EDTA complexes. II. Relation between the dissociation of complex anions and the adsorbability on ion-exchange resin at various pH values.** Tomitsugu Taketatsu (Chem. Dept., Fac. of Sci., Kyushu Univ., Hakozaki, Fukuoka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (1), 148–151.—The amount of the metal ion ($\simeq 0.01$ M) adsorbed on Amberlite IR-112 ($NH_4$ form) in the presence of EDTA increases with decrease in pH. The pH value above which no cationic exchange of a given metal ion is observed (*e.g.*, 9·0 for Ba, 8·0 for Mg, 5·8 for Ca, 3·3 for Pb) decreases with increase in formation constant of the metal - EDTA complex. The separation of similar ions, such as that of Bi from Zn, Cd and Pb, or that of Fe from Mn, Al and Be, can be effected by the use of a cation-exchange resin at a suitable pH.

**III. Separation of bismuth from zinc, cadmium and lead.** Tomitsugu Taketatsu. *Ibid.*, 1957, **78** (1), 151–153.—Anionic EDTA complexes of Zn, Cd and Pb dissociate at pH 1·3 to 1·5, whereas that of Bi remains unchanged even at pH $< 1$. A mixed soln. of these metal ions (0·01 M each), containing a small excess of EDTA with respect to Bi, is made pH 1 with dil. $HNO_3$ and passed through a column of Amberlite IR-112. The eluate contains only the bismuth complex of EDTA. The column is washed with dil. $HNO_3$ of pH 1 and eluted with 3 N HCl (100 ml) to collect Zn and Cd; Pb is subsequently eluted with 2% ammonium acetate soln. (100 ml).                    K. SAITO

**3531. Use of complexometric analytical procedures in the iron-works laboratory.** H.-L. Wengler and W. Ausel (Klöckner-Hüttenwerk Haspe A.-G.). *Arch. Eisenhüttenw.*, 1957, **28** (1), 7–12.—In this Report No. 236 of the Chemical Committee of the Vereins Deutscher Eisenhüttenleute, the principles and applications of complexometric analysis are discussed, and procedures used in iron works for the analysis of some minerals and refractories are presented.                    G. BURGER

**3532. The high-frequency titration of acids.** V. M. Gorokhovskiĭ and Yu. Yu. Samitov. *Uch. Zap. Kazansk. Univ.*, 1956, **116** (5), 97–102; *Ref. Zhur., Khim.*, 1957, Abstr. No. 19,492.—Studies are made of the applicability of $Q$- and $F$-meters for the high-frequency titration of a series of inorganic and organic acids in low concn., and the possibility of the step titration of polybasic acids. In titration with the $Q$-meter (at 100 megacycles) it is established that the titration curve of a mixture of HCl and acetic acid (2 to 4 ml of 0·01 N HCl and 2 ml of 0·01 N acetic acid) has two inflections, corresponding to the neutralisation of $H^+$ from the HCl and from the acetic acid, respectively. In determining the concn. of $HCO_3^-$ in natural waters, a V-shaped curve is obtained, with a sharp inflection at the equivalence point. The relative error is 0·2 to

0·5% (in titration with methyl orange it is 0·5 to 1·5%). The *F*-meter (at 26 megacycles) has been used for the titration of $H_2SO_4$ (at concn. $< 0.01$ g-equiv. per litre) with alkali ($\simeq 3 \times 10^{-3} N$).

C. D. KOPKIN

**3533. Quantitative aspects of microchromatography.** S. H. Ven Horst, V. Jurkovich and Y. Carstens (Dept. of Chem., Marycrest Coll., Davenport, Iowa, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 788–791.—The various contributions of variables in a paper-chromatographic determination of amino acids are critically examined. Of particular interest are variations due to paper blanks, technique of location with ninhydrin, and the effects of heat, light and humidity.

D. A. PANTONY

**3534. Quantitative chromatography on treated paper. VI. Effect of pH of the sample solution on the area of chromatograms of anions.** Akira Murata (Fac. of Engng, Shizuoka Univ., Oiwake, Hamamatsu). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (1), 57–62.—The areas of the chromatographic spots of $MoO_4^{2-}$, $WO_4^{2-}$, $VO_4^{3-}$, $CrO_4^{2-}$, $Cl^-$, $SeO_4^{2-}$, $PO_4^{3-}$ and $SeO_3^{2-}$ were measured at various pH values of the soln., on filter-paper impregnated with alumina (*cf. Anal. Abstr.*, 1955, **2**, 1443). The areas of the spots of those anions that remain unchanged with change in pH are const. over a wide range of pH (*e.g.*, pH 3 to 10 for $Cl^-$), whilst the spots of those anions that produce multiple ions at a lower pH value decrease in area with decrease in pH. A similar trend is observed for weak acids, presumably owing to the smaller extent to which ionisation takes place at a low pH value. At pH values $> 10$, the area of all anions becomes greater, and at $<$ pH 3 it becomes smaller, and it would seem that the nature of the treated paper changes at these pH regions.

**VII. Determination of chromate, molybdate, tungstate and vanadate ions.** Akira Murata. *Ibid.*, 1957, **78** (3), 395–400.—Since the areas of the chromatographic spots of $CrO_4^{2-}$, $MoO_4^{2-}$ and $WO_4^{2-}$ remain unchanged at pH 7 to 10, their determination can be satisfactorily carried out on treated paper. The area is proportional to concn. for 50 to 200 $\mu g$ of $CrO_4^{2-}$, 50 to 250 $\mu g$ of $MoO_4^{2-}$, and 100 to 400 $\mu g$ of $WO_4^{2-}$. Vanadate is present in a condensed form at a pH $< 10$ and the area - pH diagram fails to give a plateau in a weakly basic region; hence the pH value of the soln. must be kept strictly constant. The separation of these anions from others has been studied and the allowable limits of foreign ions are listed. Separation of these anions from each other and from $SO_4^{2-}$ is difficult, and it is likely that the formation of the chromatogram is due to the exchange of these anions with $ClO_4^-$, used for the preparation of the treated paper.

K. SAITO

**3535. Fundamental studies on paper chromatography. II. Effect of temperature and the shape of the paper on the *R*$_F$ value of several metal ions.** Shori Harasawa and Tomikichi Kinami (Fac. of Engng, Yamaguchi Univ., Ube, Yamaguchi Prefecture). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (1), 96–100.—The chromatographic separation of Cu, Bi, Cd and Hg, of Fe[III], Co and Ni, and of Li, Na and K was studied under various conditions. The $R_F$ values of metal ions increase with increase in temperature to an extent that varies with the developing agent; no significant influence of temp. was observed, however, at about room temp. The width of the paper has little effect on

the $R_F$ value, provided that it is uniform. When the upper part is narrower than the lower part, the $R_F$ value decreases, despite a rapid rise of the developing agent, presumably owing to insufficient ascent of the solute. On the other hand, the $R_F$ value is greater when the paper is narrower at the bottom than at the top.

K. SAITO

**3536. Rapid paper-chromatographic analysis of group 1 cations.** S. P. Marion and J. Psihas (Brooklyn Coll., New York). *J. Chem. Educ.*, 1957, **34** (2), 87.—Strips (150 mm $\times$ 15 mm) of filter-paper are fitted into 6-in. test-tubes and standards are prepared with 0·05 $M$ test solutions of Ag, Pb and Hg[I]. The strips are immersed in 0·025 $M$ $K_2CrO_4$ solution and then exposed to $NH_3$ vapour, colours and colour changes being noted after each operation. The strip from a given sample under examination is compared with the standards. An uppermost area, yellow after development with $K_2CrO_4$ and orange after exposure to $NH_3$, indicates Pb. Directly under this zone is a zone of Hg[I], which is black after development with $NH_3$. The lowest zone, brick-red after treatment with $K_2CrO_4$, fading to yellow with $NH_3$, indicates Ag.

S.C.I. ABSTR.

**3537. Gas-adsorption chromatography with thermal conductivity indication.** L. Sokol (Stalinovy závody n.p., Záluží, Czechoslovakia). *Chem. Průmysl*, 1957, **7** (4), 189–190.—The gas chromatography of $C_1$ to $C_5$ hydrocarbons on alumina, with hydrogen as carrier gas, is described. The eluted components are detected by thermal conductivity and automatically recorded by a millivoltmeter (0 to 10 or 0 to 50 mV). The composition of the analysed mixture can be directly computed from the area of the individual elution curves.

J. BÖSWART

**3538. Combined use of ion exchangers and chelating agents. Preliminary communication.** L. Wünsch (Res. Inst. Inorg. Chem., Ústí nad Labem, Czechoslovakia). *Chem. Listy*, 1957, **51** (2), 376–378.—A new method of elution ion-exchange chromatography is described. Cations can be selectively eluted from a cation-exchange column by means of a volumetric soln. of EDTA (disodium salt) or Chenta reagent, sometimes in combination with other complexing agents. The unchanged EDTA in the eluate can be titrated. This method was demonstrated by the determination of Ca in the presence of Mg. The mixture of Mg and Ca is added to a column of Permutit RS (length 20 cm, diameter 1 cm), the column is eluted first with the standard EDTA soln. buffered at pH 6·1 for Ca, and then with the same soln. buffered at pH 9 for Mg. After each elution, the unchanged EDTA in the eluate is titrated.

J. ZÝKA

**3539. Graphical methods for determining high concentrations by spectrographic analysis.** L. I. Topalov (Zaporozhe Ferrous Alloy Works). *Zavod. Lab.*, 1957, **23** (5), 549–556.—Methods of constructing calibration curves for binary, ternary and multi-component systems, so that the accuracy of spectrographic determination of high contents of metals in alloys is increased, are described.

G. S. SMITH

**3540. Precision and accuracy of spectrophotometric analysis of binary systems. III. Relation between the precision of a determination and the concentration of the determined components.** J.

Świętosławska and H. Walędziak (Dept. of Technical Physics, Inst. of General Chemistry, Warsaw). *Chem. Anal., Warsaw*, 1956, **1** (4), 246–254.—Results are presented of studies carried out in order to determine relative errors when employing a spectrophotometric method for the estimation of carbazole (at 292 m$\mu$) and anthracene (at 332·5 m$\mu$) in the presence of each other. The precision of results obtained for carbazole was practically constant in the concn. range 100 to 30%, slowly decreased between 30 and 10%, and deteriorated rapidly at 10% or below. For anthracene, the precision was nearly constant between 100 and 80% and similar to that for carbazole concn. between 100 and 30%. From 80 to 20% there was a steady fall in precision and below 20% there was a far more rapid deterioration in precision than in the determination of carbazole at the same concn. These results showed good agreement with theoretical calculations except for anthracene concn. below 10%.　　　　　K. F. Sporek

**3541. Volumo-colorimetry and its applications in chemical analyses.** A. Ionesco-Matiu (Fac. de Pharmacie, Bucharest). *Chim. Anal.*, 1957, **39** (5), 175–179.—The method of volumo-colorimetry involves quant. development of colour by a redox reaction followed by titration with a reagent that causes decolorisation. A review is given of applications of the method to the analysis of biochemical, pharmaceutical and industrial materials.
　　　　　A. R. Rogers

**3542. Main tendencies in the growth of polarographic analysis.** J. Heyrovský (Polarographic Inst., Czech. Acad. Sci.). *Zavod. Lab.*, 1957, **23** (4), 399–409.—A review with 48 references.
　　　　　G. S. Smith

**3543. Adsorption by falling particles and its use in the analysis of very dilute solutions [and gases].** V. B. Aleskovskiĭ. *Trudȳ Leningr. Tekhnol. Inst. im. Lensoveta*, 1956, (35), 133–137; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,676.—The sample of adsorbent in the form of very small particles is introduced into the soln. or gas. The particles fall or rise with const. velocity, passing through the whole of the fluid, and adsorbing the substance being determined. An instrument is described for adsorption by falling particles, which may be used for the determination of the concn. of $Cu^{2+}$ in aq. soln.　　　　　C. D. Kopkin

**3544. Chemical analysis of surfaces by nuclear methods.** S. Rubin, T. O. Passell and L. E. Bailey (Stanford Res. Inst., Menlo Park, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 736–743.—High-energy proton or deuteron bombardment produced by the High Voltage Engineering Corp., Model A.H., 2,000,000-volt Van de Graaf accelerator, incorporated in the apparatus described, has been used in the analysis of solid surfaces. The theory of the nuclear scattering method, and determinations of O, Al, Si, S, Ca, Ag, Fe, Cu, Ba and Pb by the scattering method are detailed; C, N, O, F and Na were also determined by methods other than scattering. Momentum profiles are shown, and scattering analysis and pulse-height spectra included. All elements should be detectable to a depth of several microns, with sensitivity in the range $10^{-3}$ to $10^{-6}$ g per sq. cm.　　　E. G. Cummins

## 2.—INORGANIC ANALYSIS

**3545. General method for the rapid determination of total strong acid anion in solution.** M. J. Cabell (A.E.R.E., Harwell, England). A.E.R.E. Report C/M 155, 1957, 6 pp.—The method consists in passing a salt soln. through a column of resin, whereby the cations displace an equiv. number of protons, these being estimated by titration. Results are given for thorium, calcium and sodium soln., with Zeo-Karb 225 as the resin. The applicability of the method for determining total nitrate in plutonium nitrate soln. is discussed.
　　　　　W. J. Wright

**3546. Fast separations of cations using ascending paper chromatography.** G. W. Warren and R. W. Fink (Dept. of Chem., Univ. Arkansas, Fayetteville, U.S.A.). *J. Inorg. Nuclear Chem.*, 1956, **2** (3), 176–179.—With 5 to 10 $\mu$g of the cations it has been shown that separations can be achieved in 2 to 20 min. when the development is carried out in an atmosphere that is supersaturated with solvent. The results of experiments with $Pd^{2+}$, $Ag^+$, $Cd^{2+}$, $Fe^{2+}$, $Fe^{3+}$, $Co^{2+}$, $Ni^{2+}$, $Cu^+$, $Cu^{2+}$, $Bi^{3+}$, $Pb^{2+}$, $Tl^+$, $Tl^{3+}$, $Hg^+$, $Hg^{2+}$, $Au^{3+}$ and $Pt^{4+}$ and ten developers (eight of which contain at least 80% of acetone as solvent) are tabulated.　　　G. J. Hunter

**3547. Electro-migration on paper in the separation of ions. II.** Hari Gopal Mukerjee (Scottish Church Coll., Calcutta, India). *Z. anal. Chem.*, 1957, **155** (4), 267–271 (in English).—The migration sequences of the ions of As, Sb and Sn, their separation from each other and from other ions such as those of Ag, Pb, Hg, Cu, Bi, Cd, Zn, Co and Ni in the presence of various electrolytes of different strengths have been studied. Successful separations of cations with distinct separation zones have been achieved from ternary mixtures of the following types: As - Sb - Bi, As - Pb - Zn, Sb - Cd - Zn, As - Bi - Co, As - Cd - Co, As - Pb - Co and As - Cd - Ni with 0·1 $N$ Na citrate; As - Sb - Sn with 0·5 $N$ Na citrate; As - Sb - Ag, As - Hg - Bi and Sb - Ag - Pb with Na K tartrate; As - Hg - Bi with EDTA (disodium salt); As - Hg - Zn with $NaNO_2$; As - Hg - Pb and As - Hg - Zn with $NH_4SCN$; Sb - Ag - Zn, Sb - Ag - Co and Sb - As - Co with $H_3PO_4$; and As - Bi - Cd with $HNO_3$. The observations of Strain and Sullivan (*Anal. Chem.*, 1951, **23**, 816) that $Sn^{2+}$ form a large number of separate distinct zones in many electrolytes is confirmed.

**III.** Hari Gopal Mukerjee. *Ibid.*, 1957, **155** (6), 406–411 (in English).—A description is given of the paper electrophoresis of the ions of Ag and Hg and of their separation from each other and from the ions of Pb, Cu, Bi, Cd, Co and Ni in different electrolytes. Mixtures containing as many as five cations have been effectively separated.
　　　　　D. F. Phillips
　　　　　A. R. Rogers

**3548. Titrations with potassium cyanide.** J. Doležal, V. Simon and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (5), 880–883.—A soln. of KCN is a suitable volumetric reagent for the titration of Cu and Ni in aq. $NH_3$ soln., with murexide as indicator. The indirect micro-determination of Ni, $Hg^{II}$, Ag, $Au^{III}$ and Pd can be carried out by adding an excess of KCN soln., the excess of which can be determined with $NiSO_4$ soln., with the same indicator. For the direct titration of Cu (0·01 $M$), only one drop of aq. $NH_3$ (for 50 ml of the soln.)

must be added; for the titration of Ni, the addition of 1 to 2 ml of conc. aq. $NH_3$ is necessary. *Procedure for indirect titrations* ($Ni$, $Hg$, $Au$, $Ag$, $Pd$)— To the neutral or slightly acid soln. of the sample add a known excess of KCN soln. and conc. aq. $NH_3$ (1 to 2 ml), dilute with twice-distilled $H_2O$ to 50 ml, add murexide indicator and titrate with a volumetric soln. of $NiSO_4$ till the violet colour changes to yellow. In the titration of Hg, a slight excess of KCN must be used; for the determination of Au, the back-titration must be carried out immediately after the excess of KCN has been added. The possibilities of the determination of Hg in pharmaceuticals, Ag in AgCl, Pd in the presence of Ir and Rh, and Hg or Ni in the presence of Zn are discussed. As little as 25 $\mu$g of Pd and 150 $\mu$g of Ag (Au) were determined by the described procedure.                    J. ZÝKA

**3549. The use of complex formation for the separation and determination of elements by an extraction method.** I. P. Alimarin and I. M. Gibalo. *Vestnik Moskov. Univ.*, 1956, (5), 55–59; *Ref. Zhur., Khim.*, 1957, Abstr. No. 8369.—Acetylacetone (**I**) and EDTA (disodium salt) (**II**) give complexes of different stability with different elements. The acetylacetone complexes, in contradistinction to the EDTA complexes, are easily extracted with organic solvents, *e.g.*, $CCl_4$. The behaviour of the acetylacetone complexes of Fe, Be, Cd, Co, Ni, Mn, Cu, Pb and Zn on extraction with $CCl_4$ in the presence of **II** has been studied. Beryllium is completely extracted at pH 9. To separate Be from Fe, to 15 ml of soln. (pH 2 to 3) containing $BeSO_4$ and $FeCl_3$ in a separating funnel add 15% **I** soln. (5 ml), 0·05 $M$ **II** soln. (7 ml), two drops of conc. aq. $NH_3$ and 7 ml of $CCl_4$. Shake for 5 min. and separate the organic phase; repeat the extraction twice more, adding the same reagents with the exception of **II**. To the organic phase add water (20 ml) and HCl (15 ml), evaporate the water layer to half vol., precipitate Be with aq. $NH_3$ and determine gravimetrically as BeO. The Be is practically completely extracted; the Fe is not extracted. To separate small quantities of Co from Fe, neutralise the soln. with aq. $NH_3$ to a pH of 3 to 4, add 0·05 $M$ **II** soln. (8 to 10 ml), 15% **I** soln. (5 to 6 ml), conc. aq. $NH_3$ (2·5 ml) and $CCl_4$ (7 ml), and shake in a separating funnel for 3 to 4 min. Repeat the extraction twice more, adding the same quantities of **I** and $CCl_4$. This method will also free Fe completely from Cu, Ni, Pb, Cd, Zn and Mn.                    C. D. KOPKIN

**3550. Use of Trilon B [EDTA (disodium salt)] in ion-exchange chromatography. I.** R. N. Golovatyĭ. *Dopovidi ta Povidomlennya, L'vivs'k. Univ.*, 1955, **6** (2), 131–134; *Ref. Zhur., Khim.*, 1957, Abstr. No. 11,992.—For the separation of Fe from Mn, Zn, Be, Mg, Ba, Ca, Li and K, add to the weakly acid soln. two or three drops of a 5% soln. of $NH_4SCN$ and 0·2 $N$ EDTA (disodium salt) (**I**) till the soln. is colourless. Neutralise to methyl orange with aq. $NH_3$ and pass the soln. through a layer of cationite (H form) (**II**) (12 g) at a rate of 1 to 2 ml per min.; Fe passes into the eluate. For the separation of Al from Be, Mg, Mn, Zn and Li, add to the soln., weakly acid in HCl, a two- or three-fold excess of **I** (based on Al), neutralise with aq. $NH_3$ to methyl orange and pass the soln. through a layer of **II**; Al passes into the eluate. At pH 4·4, Cr is quant. adsorbed by **II** from aq. soln. in the presence of any excess of **I**; this is used in the

separation of Cr from Al, Fe, Ni and Cu. To separate Ni from Mg, Be, Mn and Zn, add to the weakly acid soln. a two- or three-fold excess of **I** (based on Ni), neutralise with aq. $NH_3$ to methyl red and pass the soln. through a cationite ($NH_4$ form); Ni passes into the eluate. To remove Cu from Ag, add aq. $NH_3$ to the soln., followed by a saturated soln. of **I** till the blue colour is discharged. When the soln. is passed through a layer of cationite ($NH_4$ form), Cu passes into the eluate, while Ag is held by the cationite.                    C. D. KOPKIN

**3551. Analytical studies on masking reactions. I. Masking reactions of aluminium, ferric and titanic ions by means of sulphosalicylic acid.** Masayoshi Ishibashi, Takumi Tanaka and Tamotsu Kawai (Sakai Chem. Ind. Co. Ltd., Ebisujima, Sakai, Osaka). *Japan Analyst*, 1956, **5** (11), 609–613.— Ferric iron remains in the aq. soln. after heating for 1 hr. at >80° in a weakly alkaline soln. containing sulphosalicylic acid (**I**) (>10 times the equiv. of the Fe). Quadrivalent Ti does not undergo basic hydrolysis at room temp. in the presence of **I**, but undergoes hydrolysis when heated at >30°. For the pptn. of Al, Fe and Ti from the masked soln., the formation of oxinate, sulphide and hydroxide, respectively, is recommended.

**II. Masking of manganous, magnesium and cupric ions by means of sulphosalicylic acid.** Takumi Tanaka. *Ibid.*, 1956, **5** (11), 613–616.—Manganese is masked in an alkaline soln. (pH >9) in the presence of an equiv. amount of **I** and a reducing agent such as hydroxylamine hydrochloride (>3 times the equiv. of the Mn). Pptn. of the hydroxides of Cu and Mg is suppressed at pH <11 in an NaOH soln. and at a higher pH in an ammoniacal soln. in the presence of **I** (>3 times the equiv. of the metal ion). The masked ions are recovered by the pptn. of their oxinates, or by pptn. as magnesium phosphate or copper thiocyanate.

**III. Masking of cadmium, zinc and nickel ions by means of sulphosalicylic acid.** Takumi Tanaka. *Ibid.*, 1956, **5** (12), 673–676.—Cadmium does not undergo alkaline hydrolysis at pH <8 in the presence of more than twice the equiv. of **I**. The Zn - **I** complex is unstable in an alkaline soln., undergoing hydrolysis at pH >7. The Ni - **I** complex is not hydrolysed, and remains unchanged on being boiled in the presence of >3 times the equiv. of **I**. For the pptn. of the masked ions, the oxinate and the sulphide method can be used, or Cd can be quant. pptd. as carbonate, Zn as phosphate and Ni as phosphate or the dimethylglyoxime complex.

**IV. Masking of cobaltous, chromic and indium ions.** Takumi Tanaka. *Ibid.*, 1956, **5** (12), 677–679. —Chromic ions are masked with **I** in an alkaline soln. only in the presence of $NH_4^+$, whereas $Co^{3+}$ and $In^{3+}$ remain in the soln., in the presence of, respectively, >40 times and >5 times the equiv. of **I**, on boiling at pH <10. In a more basic soln. the complexes are unstable. Bivalent Co is quant. pptd. from the masked soln. with nitrosonaphthol or oxine, and In with oxine.

**V. Separation of aluminium and ferric iron from titanium by the use of sulphosalicylic acid.** Takumi Tanaka. *Ibid.*, 1957, **6** (1), 13–16.—These findings were applied to the separation of Ti from Al and Fe. A basic soln. containing <1 g each of Al and Ti is made pH >9·4 in the presence of **I** (>10 times the weight of the metal ions), and boiled to precipitate Ti; Al is pptd. as oxinate in the filtrate. In the presence of Al > Ti, Ti should be re-pptd. A basic soln. of pH 8 to 9 containing both Fe and Ti (< 0·1 g per 100 ml) masked with **I** is treated with $H_2S$

to precipitate Fe as sulphide, and Ti in the filtrate is hydrolysed by boiling. In the presence of Ti > Fe, the iron sulphide should be re-pptd.

**VI. Separation of iron and magnesium from aluminium by the use of sulphosalicylic acid.** Takumi Tanaka. *Ibid.*, 1957, **6** (1), 16–19.—A basic soln. of pH 9 containing Fe and Al (both < 0·1 g) masked with **I** is treated with $(NH_4)_2S$ to precipitate Fe as sulphide, and Al is pptd. as oxinate in the filtrate. The latter pptn. is not affected by $S^{2-}$. Magnesium and Al (both < 0·1 g) are pptd. in an acid and a basic soln., respectively, with oxine. Marked adsorption was observed on pptn. of their phosphates in a soln. containing **I**.

**VII. Analysis of ilmenite with sulphosalicylic acid.** Takumi Tanaka. *Ibid.*, 1957, **6** (2), 78–80.—The determination of Ti, Fe, Al and Mn in ilmenite and intermediates for the preparation of titanium white was carried out by the method described above. Ilmenite (0·5 g) is fused with $K_4P_2O_7$ (5 g), the melt is leached with 5% $H_2SO_4$, and filtered, and the residue is ignited and weighed as $SiO_2$. A 50-ml portion of the filtrate (250 ml) is treated with $NH_4Cl$ (5 g), **I** (5 g) and 6 N aq. $NH_3$ to produce 100 ml of soln. of pH 8 to 9. The sulphides of Fe and Mn are pptd. with $H_2S$ and separated from each other with oxine. Titanium is pptd. by boiling the filtrate which has been adjusted to pH >9 with aq. $NH_3$ soln.; Al is pptd. as oxinate. The separation of Ti from Al is thus more easily achieved than by the use of tartrate. The latter method appears to give a slightly high result for Ti.

**VIII. Complex formation between sulphosalicylic acid and ferric iron.** Masayoshi Ishibashi, Takumi Tanaka and Tamotsu Kawai. *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (10), 1603–1606.—The composition and the dissociation const. of the Fe - **I** complex were determined with the aid of polarography and spectrophotometry. The half-wave potential changes with pH, indicating the presence of two different complexes at pH >6 and < 4. The stable complex formed at pH >6 appears to have a molar ratio of 1:3 (Fe: **I**); the dissociation const. is $2·5 \times 10^{-11}$ at 25°.

**IX. Complex formation between sulphosalicylic acid and titanium.** Masayoshi Ishibashi, Takumi Tanaka and Tamotsu Kawai. *Ibid.*, 1956, **77** (10), 1606–1609.—Similar experiments were carried out with $Ti^{4+}$. Two complexes are formed, at pH >8, and pH 1 to 7, the ratio of Ti to **I** being 1:2 and 1:3, respectively. The former is less stable than the ferric complex, the dissociation const. being $1·2 \times 10^{-9}$ at 25°.

**X. Complex formation between sulphosalicylic acid and nickel.** Masayoshi Ishibashi, Takumi Tanaka and Tamotsu Kawai. *Ibid.*, 1956, **77** (10), 1609–1612.—The formation of complexes of Ni with **I** was studied by absorption spectroscopy and compared with that of the nickel ammine complex. The formation is most marked at pH ≃ 9. Polarography showed that, at pH < 5, no appreciable complex formation occurs. It is likely that, in an ammoniacal **I** soln., some of the $NH_3$ groups in $Ni(NH_3)_6^{2+}$ are replaced with **I**.

**XI. Complex formation between sulphosalicylic acid and manganese.** Masayoshi Ishibashi, Takumi Tanaka and Tamotsu Kawai. *Ibid.*, 1956, **77** (10), 1613–1615.—An experiment similar to that for Ni was carried out with $Mn^{2+}$ in the presence of hydroxylamine hydrochloride. No appreciable complex formation takes place at pH < 9. The complex formed at pH >10·5 has a molar ratio of 1:2 (Mn: **I**), and the dissociation const. is ≃ $10^{-12}$.

K. SAITO

**3552. The masking of ions for the simultaneous polarographic determination of elements with coincident reduction potentials.** F. K. Baev and P. R. Kovalenko. *Trudy̆ Komiss. Anal. Khim., Akad. Nauk SSSR*, 1956, **7** (10), 119–135; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,003.—The use of the simultaneous polarographic determination of elements with coincident reduction potentials has been investigated from the point of view of increasing the specificity by masking one of the ions being reduced. Masking of the $Sn^{4+}$ was used in order to attain a direct polarographic determination of Sn and Pb when both metals are present in a soln. containing large amounts of Zn. The Pb is determined after suppression of the diffusion current due to Sn, and the Sn by the difference of the heights of the polarographic waves obtained before and after the masking of the $Sn^{4+}$. In the presence of large quantities of Zn (50 to 200 g per litre) and with a concn. of 0·005 to 1 g of Sn per litre, the diffusion current of the Sn is completely suppressed by a concn. of 0·05 to 1% of citrate ion at pH 1·5 to 3·5. Under these conditions Zn is not reduced, and the height of the polarographic wave due to Pb is directly proportional to its concn. In HCl soln., the total height of the diffusion waves of $Sn^{4+}$ and Pb is equal to the sum of the heights of the waves of these metals taken separately. There exists a direct proportionality between the concn. and the wave height both for Sn and Pb.        C. D. KOPKIN

**3553. Use of oscillographic polarography in quantitative analysis. VII. Micro-determination of some metals.** R. Kalvoda (Polarogr. Inst., Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (4), 696–703.—For the identification and determination of depolarisers in the concn. range $10^{-5}$ to $10^{-9}$, the method of preliminary electrolysis of the soln. to be determined, in connection with oscillographic analysis of the amalgam thus formed, was used. The transition of the element into the soln. by anodic polarisation shows, by oscillographic registration of the relation $dV/dt = f(V)$, typical kinks, the depth of which depends on time. The conditions for analytical utilisation of these phenomena for the determination of Cu, Sn, Bi, Cd, In, Pb, Tl, Zn, Ba, Sr, Na, K and $NH_4^+$ alone, or in the presence of elements which do not form amalgams, are discussed.        J. ZÝKA

**3554. Use of radioactive isotopes for analysis of certain mixtures [*e.g.*, mixtures of $CaSO_4$ and $Na_2SO_4$].** V. F. Fursenko (Rostov Inst. of Railway Engineers). *Zavod. Lab.*, 1957, **23** (4), 410–411.—The use of two labelled elements for analysing a binary mixture, *e.g.*, $^{35}S$ and $^{45}Ca$ for $CaSO_4$ - $Na_2SO_4$ mixtures, is described. If $I_1$ and $I_2$ are the $\beta$-radiation intensities of the isotopes as measured by a counter, the total intensity $I = I_1 + I_2$, and if $I'$ is the total intensity when measurements are made through a filter, *e.g.*, aluminium foil, then $I' = k_1I_1 + k_2I_2$, where $k_1$ and $k_2$ are coeff. of radiation weakening of the two isotopes by the filter. These coeff. can be determined experimentally for each isotope separately. By solving the equations, $I_1$ and $I_2$, and hence the concn., can be determined from the values found experimentally for $I$ and $I'$. The results with $CaSO_4$ - $Na_2SO_4$ mixtures show that the method gives only an approx. indication of the concn.

G. S. SMITH

**3555. Separation of hydrogen, oxygen, nitrogen, methane and carbon monoxide by gas-adsorption chromatography.** G. Kyryacos and C. E. Boord (Dept. of Chemistry, Ohio State University,

Columbus, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 787–788.—Separation of these gases is achieved in a stream of He by use of a gas-chromatographic technique, in which a molecular sieve base is employed, with thermal conductivity detection. The technique is applied to analysis of hydrocarbon flame products. D. A. PANTONY

**3556. The determination of traces of moisture in gases.** M. Šingliar and J. Zubák (Res. Inst. Acetyl. Chem., Nováky, Czechoslovakia). *Chem. Průmysl*, 1956, **6** (10), 426–427.—The method, depending on reaction with magnesium nitride and colorimetric determination of the $NH_3$ formed by means of Nessler reagent, is capable of determining $5 \times 10^{-5}$ % (w/v) of $H_2O$. The extinction is measured at 470 m$\mu$. J. BÖSWART

**3557. Methods for the determination of water.** Z. Łada (Anal. Dept., Inst. of General Chemistry, Warsaw). *Chem. Anal., Warsaw*, 1956, **1** (4), 235–245.—A survey is presented of physical, physico-chemical and chemical methods for the determination of moisture in a wide range of materials. The Fischer reagent is considered to be the most satisfactory and most widely applicable for routine work. (46 references.) K. F. SPOREK

**3558. Thermogravimetric pyrolysis of some metal tetraphenylboron compounds.** W. W. Wentlandt (Dept. of Chem., Texas Technol. Coll., Lubbock, U.S.A.). *Chemist Analyst*, 1957, **46** (2), 38–39.—Tetraphenylboron compounds of $NH_4$, K, Rb and Cs have been found to possess good thermal stability. Other ions, *e.g.*, $Ag^+$, $Hg^+$ and $Hg^{2+}$, also form insol. ppt. with the tetraphenylboron ion, and the thermal stabilities of the resulting compounds, as well as those of Na and Li, have been studied on the thermobalance. The curves are shown and discussed. Sodium and $Hg^{II}$ tetraphenylboron are the most stable and Ag tetraphenylboron the least stable. W. J. WRIGHT

**3559. Polarographic method for the determination of the ratio of alkali metals (sodium to potassium) without previous chemical separation.** A. Duca (Chem. Inst. Acad., Cluj, Romania). *Acad. Rep. Populare Romîne, Studii Cercetări Chim.*, 1956, **4**, 131–144.—The slight difference between the reduction voltages of K and Na permits only the polarographic determination of their sum. By increasing the concn., first of one, then of the other metal by a known amount, two new polarographic points are obtained from which the ratio of the two components can be calculated. The optimum conditions for a large difference between the heights of the polarographic waves were established by studying the effect of dropping time, temp., ratio of alkali metals to soln., type of base soln. (LiOH or tetramethylammonium hydroxide) and recording conditions. The determination of the ratio up to 1:10 or even 1:50 was found feasible. CHEM. ABSTR.

**3560. Spectrographic analysis of lithium, strontium and barium for impurities.** M. A. Notkina and S. M. Solodovnik. *Zavod. Lab.*, 1957, **23** (5), 569–572.—Arc methods are described for determining a number of impurities in Li, Sr and Ba. G. S. SMITH

**3561. Spectrographic determination of sodium in potassium hydroxide.** N. Ya. Ugnyachev and T. I. Tyutyunnikova. *Trudy Vses. Inst. Sod. Prom.*, 1956, **9**, 113–116; *Ref. Zhur., Khim.*, 1957, Abstr.

No. 15,730.—To determine Na within the range 0·005 to 2%, introduce the soln. of sample or standards into the cavity in the lower carbon electrode, which has not been cooled after an ignition. Excite the spectrum in an a.c. arc discharge with a copper upper electrode with a gap of 1·5 mm and current strength 4 amp. The error of the analysis is $\pm 10$%. To determine Na within the range 0·4 to 8%, transfer drops of the soln. to the cooled flat end of the carbon electrode. The analytical lines are Na 3302·3 and K 3446·7 A. The error is $\pm 5$%. C. D. KOPKIN

**3562. Qualitative examination and determination of potassium with sodium tetraphenylboron.** M. Muraire (Laboratoire S.N.C.F.). *Chim. Anal.*, 1957, **39** (5), 184–188.—Precise conditions are described for the detection of $K^+$ in the presence of ammonium salts and for the determination of 10 to 40-mg quantities of K with a precision of $\pm 0·2$%. A. R. ROGERS

**3563. Determination of alkali metals: sodium - caesium, potassium - caesium (without chemical separation) by a derivative polarographic method.** A. Duca and N. Buruleanu (Chem. Inst. Acad., Cluj, Romania). *Rev. Chim., Bucharest*, 1956, **7** (7), 430–432.—By adopting a method analogous to that used for Na - K (*cf.* Duca, *Anal. Abstr.*, 1957, **4**, 3559), the authors obtained derived polarographic waves of different heights and depths due to different coefficients of diffusion of equal quantities of Na, K and Cs ions. Initially graphs are drawn from soln. of identical concn. of the salts in question, and from the difference between maxima and minima of the waves the proportions of the two metals present in an unknown soln. are calculated. H. SHER

**3564. Determination of a small quantity of copper by the volumetric iodate method.** I. L. Bagbanly. *Meruzeler AzerbSSR Elmler Akad. Dokl. Akad. Nauk AzerbSSR*, 1956, **12** (9), 639–642; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,735.—The method described is based on the oxidation of $SCN^-$ and $Cu^+$ in the compound $Cu[Cr(SCN)_4(NH_3)_2]$ with $KIO_3$ in acid medium. The method may be used for the determination of Cu in steel. Decompose the sample by warming on the water bath with 20 ml of $H_2SO_4$ (1:4), filter, evaporate the filtrate to 10 ml, add 10 ml of a freshly prepared 2·5% soln. of Reinecke's salt and warm on the water bath until the ppt. has coagulated and the liquor is clear (15 min.). Filter off the ppt., wash it with hot water and decompose it with 5% NaOH soln. Acidify with HCl and titrate with $KIO_3$ soln. Up to 0·008 mg of Cu can be determined in the presence of several other cations with satisfactory accuracy. The time for a determination is $\geqslant 1$ hr. C. D. KOPKIN

**3565. The quantitative determination of copper by precipitation chromatography.** K. M. Ol'shanova and M. A. Petrova. *Trudy Moskov. Tekhnol. Inst. Myas. i Moloch. Prom.*, 1956, (6), 184–187; *Ref. Zhur., Khim.*, 1957, Abstr. No. 19,527.—Pptn. chromatography is used for the determination of microgram quantities of $Cu^{2+}$ in extracts from meat and fish conserves. The method is based on the functional relationship between the length of the coloured zone of $Cu_2Fe(CN)_6$ on an alumina column mixed with $K_4Fe(CN)_6$ soln., and the concn. of $Cu^{2+}$ in the sample. The diameter of the column is 5 mm, the length 100 mm. The adsorbent is a

mixture of 100 g of anhyd. alumina and 1 g of $K_4Fe(CN)_6.3H_2O$ dissolved in 10 ml of water. The lengths of the zones are determined visually. At pH 5, the length of the zone varies from $\simeq 1$ mm for 0·0005 $N$ soln. to 6 mm for 0·01 $N$ soln. In the presence of free acetic acid and HCl, the length of the zones is considerably increased. Alkali-metal and alkaline-earth-metal ions do not influence the length of zone, but heavy-metal cations ($Zn^{2+}$ and $Fe^{3+}$) which give poorly sol. compounds with $K_4Fe(CN)_6$ interfere. The error in the determination of $Cu^{2+}$ in extracts from conserves is $\simeq 3\cdot 3\%$.

C. D. KOPKIN

**3566. Polarographic analysis with the use of EDTA as supporting electrolyte. I. The behaviour of the metals of the copper group.** Takashi Yoshino (Fac. of Engng, Yamaguchi Univ., Ube, Yamaguchi Prefecture). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (1), 131–135.—The polarograms of $Cu^{2+}$, $Bi^{3+}$, $Pb^{2+}$ and $Cd^{2+}$ were studied in 0·1 $M$ EDTA at various pH values. The wave due to Bi or Pb is not affected at pH >7 or by the presence of anions that form insol. salts with these metals. Separation of the waves due to Bi, Cu and Pb or Cd is satisfactorily effected both in weakly acid or basic soln.

**II. Polarographic characteristics of lead in a supporting electrolyte containing EDTA.** Takashi Yoshino. *Ibid.*, 1957, **78** (1), 135–138.—The polarogram of $Pb^{2+}$ in a supporting soln. containing various amounts of EDTA, and over a range of pH values, was studied with reference to the contribution of kinetic current. The $E_{\frac{1}{2}}$ value decreases with increase in concn. of EDTA and in pH. The limiting current increases with increase in pH, and is more sensitive towards pH change in a basic soln. The use of 0·1 $M$ EDTA at pH 8 is recommended for analytical purposes, both from the point of view of linearity of the working curve and stability towards pH change.

K. SAITO

**3567. Spectrophotometric determination of copper in titanium.** A. J. Frank, A. B. Goulston and A. A. Deacutis (Watertown Arsenal, Watertown, Mass., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 750–754.—By measurement of the 452-m$\mu$ absorption of the $Cu^+$ - neocuproine complex obtained with $CHCl_3$ - ethanol extraction, the $Cu^+$ in titanium containing 0·005 to 0·40% of Cu have been determined to an accuracy of $\pm 4\%$. The only metallic interference is from $Cr^{3+}$ and methods for the removal of this interference are presented.

E. G. CUMMINS

**3568. Determination of copper by electrolysis in the presence of large amounts of iron.** S. E. Kreĭmer and N. V. Tuzhilina ("Severonikel" Combine). *Zavod. Lab.*, 1957, **23** (5), 543.—Interference of Fe in the electrolytic determination of Cu is prevented by the addition of an equivalent amount (as found by titration in the presence of salicylic acid) of EDTA.

G. S. SMITH

**3569. Separation of copper and iron on Wofatit F and Wofatit P as pyrophosphate complexes.** Z. Marczenko (Analytical Dept., Inst. for General Chemistry, Warsaw). *Chem. Anal., Warsaw*, 1956, **1** (4), 263–272.—The neutral sample soln. containing Cu and Fe is treated with an excess of $Na_4P_2O_7$ and made 1% with respect to aq. $NH_3$. The soln. is then passed through a column of Wofatit P (14 mm × 250 mm) and eluted with 100 ml of dil. aq. $NH_3$ (1:100) and 100 ml of water. Results obtained with mixtures containing 100 mg of Cu and 100 mg

of Fe indicated 99·96% retention of Cu on the resin and 99·7% elution of Fe. Wofatit F was found to be inferior to Wofatit P.

K. F. SPOREK

**3570. Use of evaporation technique for the analysis of copper and nickel for purity.** A. A. Aripov and L. M. Ivantsov (P. N. Lebedev Phys. Inst., Acad. Sci. USSR). *Zavod. Lab.*, 1957, **23** (5), 558–564.—Condensates obtained at various temp. are examined spectrographically by arc excitation, and the use of the method for analysing samples of Ni and Cu is discussed.

G. S. SMITH

**3571. Separation of copper and lead cations by ion-exchange chromatography.** A. P. Kreshkov and E. N. Sayushkina. *Trudỹ Moskov. Khim.-Tekhnol. Inst.*, 1956, (22), 116–122; *Ref. Zhur., Khim.*, 1957, Abstr. No. 8431.—The following method for separating $Cu^{2+}$ and $Pb^{2+}$ is proposed. To 50 to 100 ml of soln. containing 500-mg quantities of $Pb^{2+}$ and $Cu^{2+}$ add 3 g of tartaric acid, stir to dissolve the acid and add conc. aq. $NH_3$ (30 ml). Pass the soln. through a column of 15 g of cationite (H form) at a rate of 5 ml per min. The complex cation $[Cu(NH_3)_4]^{2+}$ and some of the $Pb^{2+}$ are adsorbed, while $[Pb(C_4H_2O_6)]^{2-}$ passes into the eluate. Wash the column with a soln. of 3 g of tartaric acid and 10 ml of conc. aq. $NH_3$ in 100 ml until all the $Pb^{2+}$ have been eluted. Evaporate the eluate and wash liquors to 150 ml and precipitate Pb with $K_2Cr_2O_7$ in the presence of Na acetate; determine the Pb in the $PbCrO_4$ by titration. Wash the cationite with 50 ml of water and $N$ $HNO_3$, add conc. $H_2SO_4$ (10 ml) to the eluate, evaporate and determine Cu iodimetrically. Another variation of the method is to pass the soln. through the cationite (H form) and wash the column with a mixture of tartaric acid and aq. $NH_3$. The Cu remains on the cationite while Pb passes into the eluate. Both methods give a good separation. Instead of tartaric acid, citric acid may be used. Similar separations are given for $Pb^{2+}$ and $Ag^+$, $Pb^{3+}$ and $Cd^{2+}$, and $Pb^{2+}$ and $Zn^{2+}$, and for the removal of $Pb^{2+}$ from $Cu^{2+}$, $Ag^+$, $Cd^{2+}$ and $Zn^{2+}$.

C. D. KOPKIN

**3572. Colorimetric determination of gold with phenols.** S. Ya. Shnaĭderman. *Izv. Kievsk. Politekh. Inst.*, 1956, **17**, 204–213; *Ref. Zhur., Khim.*, 1957, Abstr. No. 8433.—The use of phenols for the detection and determination of small quantities of Au has been studied. The sensitivity of the reaction varies from 0·5 to 10 $\mu$g in 5 ml of soln., depending on the nature of the phenol used. To determine Au, mix the test soln., containing 20 to 100 $\mu$g of $Au^{III}$, with 10 ml of a buffer soln. of pH 3 to 6. Add 1 ml of a 0·01 $M$ soln. of the phenol (phenol, catechol, resorcinol, quinol, pyrogallol, phloroglucinol, 1- or 2-naphthol or thymol) and one or two drops of a 0·5% soln. of starch to stabilise the gold sol, make up to 25 ml and measure the extinction. The content of Au is found from a calibration curve.

C. D. KOPKIN

**3573. Determination of gold by spectrophotometry in the ultra-violet region.** F. Vydra and J. Čelikovský (Inst. Anal. Chem., High-School of Chem. Technol., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (4), 768–770.—Absorption curves of halogen complexes of Au in the u.v. region were studied and the following values were obtained. For $AuCl_4^-$ in 0·1 $N$ HCl, $E_{1\,cm}^{1\,\%}$ at 311·5 m$\mu$ = 282, and for $AuBr_4^-$ in 0·1 $N$ KBr at pH 1, $E_{1\,cm}^{1\,\%}$ at 254 m$\mu$ = 2158. For analytical determinations, the concn.

4 to 30 $\mu g$ per ml in the form of $AuCl_4^-$ or 0·5 to 30 $\mu g$ per ml as $AuBr_4^-$ is recommended.

J. ZÝKA

**3574. Salicylic acid as a reagent for the spectro-photometric determination of beryllium.** L. P. Adamovich and T. U. Kravchenko (Kharkhov State Univ.). *Zavod. Lab.*, 1957, **23** (4), 416–420.—Salicylic acid and $BeOH^+$ give a complex $[BeOH(Sal)_2]^{3-}$ with an instability const. of $(4·9 \pm 0·6) \times 10^{-18}$. The optimum pH is 9·0 to 9·5, and the extinction of the soln. is best observed at 320 m$\mu$.

G. S. SMITH

**3575. Determination of beryllium in alloys and concentrates by radiometric titration.** I. P. Alimarin and I. M. Gibalo (Moscow State Univ.). *Zavod. Lab.*, 1957, **23** (4), 412–416.—The method previously described for the titration of Zr (*cf. Anal. Abstr.*, 1956, **3**, 3600) is applied to the determination of Be. The soln. (2 to 5 ml) of $BeSO_4$ containing 0·7 to 9 mg of Be is mixed with 10 ml of an acetate buffer soln. of pH 5·0 to 5·5 and 2 to 6 ml of 15% ammonium acetate soln. and titrated during energetic stirring with 0·1 $M$ $(NH_4)_2HPO_4$ containing $^{32}P$, giving an activity as measured by a counter of 20,000 to 30,000 impulses per min. per ml. At intervals during the dropwise addition of the titrant the soln. is centrifuged and the activity of 0·5 ml of the clear soln. is measured. The equivalence point is found graphically. The interference of other elements, *e.g.*, Al, Fe and Cu, can be avoided by the use of EDTA. To determine Be in bronze, the sample (0·8 to 1·3 g) is dissolved without heating in 15 ml of dil. $HNO_3$ (1 + 1), the soln. is evaporated nearly to dryness, the residue is dissolved in water, the soln. is cooled, then neutralised with 2% aq. $NH_3$, and diluted to 25 ml; a 5-ml aliquot is taken for the titration, which is carried out after addition of 25 ml of 7·5% EDTA soln., 30 ml of the acetate buffer soln., and 15 to 25 ml of 15% ammonium acetate soln. To determine Be in concentrates, the sample (0·5 to 0·7 g) (1 part) is fused with NaF (4 parts) in a platinum crucible at 1000° to 1100° for 40 to 50 min., the cooled melt is heated with conc. $H_2SO_4$ to remove Si and F, the residue is dissolved in water, the soln. is neutralised with aq. $NH_3$ and dil. $H_2SO_4$ and then diluted to 50 ml. A 5-ml aliquot is mixed with 10 to 12 ml of 7·5% EDTA soln., 25 ml of the acetate buffer soln. and 15 ml of 15% ammonium acetate soln., and the titration is carried out as described above. It is shown that in all cases the establishment of only two points on the titration curve is required.

G. S. SMITH

**3576. Stepwise EDTA titration of calcium and magnesium with C.I.202 and C.I.203 as indicators.** P. F. Lott and K. L. Cheng (School of Mines & Metall., Univ. of Missouri, Rolla, U.S.A.). *Chemist Analyst*, 1957, **46** (2), 30–31.—Calcium is directly titrated with EDTA (disodium salt) at pH 13, with C.I.202 [1-(2-hydroxy-1-naphthylazo)-2-naphthol-4-sulphonic acid] in the form of its sodium salt (Calcon) or its zinc complex (Zinchrome R) as indicator. At this pH, Mg is present as insoluble hydroxide and is not co-titrated. The pH is then adjusted to 10 and Mg is directly titrated with EDTA, with C.I.203 (Eriochrome black T) as indicator.

W. J. WRIGHT

**3577. Murexide as a reagent for the colorimetric determination of calcium.** G. G. Karanovich. *Trudŷ Vses. Nauch. Inst. Khim. Reaktivov*, 1956,

(21), 48–51; *Ref. Zhur., Khim.*, 1956, Abstr. No. 78,428.—The reaction of murexide (in aq. soln.) with $Ca^{2+}$ causes an orange - pink colour to be formed. The sensitivity at pH 6 corresponds to 20 $\mu g$ of Ca in 5 ml, and at pH 12 to 13 to 1 $\mu g$ of Ca in 5 ml. The max. colour intensity is reached after 1 min. The stability of the colour depends on the quantity of alkali added; max. stability is attained with 0·25 ml of $N$ NaOH in 5 ml. In the presence of 1 ml of $N$ NaOH in 5 ml of soln. containing 5 $\mu g$ of Ca, up to 400 $\mu g$ of $Mg^{2+}$ is allowable. It is established that $\ll 12$ $\mu g$ of $Fe^{3+}$, 1 to 3 $\mu g$ of $Cu^{2+}$, $Zn^{2+}$, $Cd^{2+}$, $Ni^{2+}$, $Cr^{3+}$ and $Mn^{2+}$, and $\gg 25$ $\mu g$ of $Hg^{2+}$ interfere. By using NaF, up to 50 $\mu g$ of $Fe^{3+}$ may be screened, but $Mg^{2+}$ interfere in this process. With a Ca to Mg ratio of 1:200, $Fe^{3+}$ and heavy metals may be complexed with diethyl-dithiocarbamate, and the coloured compounds extracted with *iso*butyl alcohol. Dilute the aq. layer to 50 or 100 ml, and in a 2- or 5-ml aliquot determine Ca with murexide at pH 12 to 13. Practically no interference is caused by the presence of Al, Pb, Ba, As, Pt and Na, in the respective ratios of Ca:M of 1:50, 1:50, 1:25, 1:12·5, 1:250 and 1:1000. The method may be used for the determination of the calcium impurity in alkali-metal salts, but the determination of Ca in ammonium salts is possible only after removal of $NH_3$.

C. D. KOPKIN

**3578. Photometric determination of calcium via precipitation of its naphthylhydroxamate.** A. M. Amin (Faculty of Engng, Univ. of Cairo, Giza, Egypt). *Chemist Analyst*, 1957, **46** (2), 31–34.—Calcium is pptd. by addition of Na naphthylhydroxamate to the boiling ammoniacal soln. The ppt. is isolated, washed by centrifuging, and dissolved with excess of EDTA (disodium salt) in the presence of an ammoniacal buffer (pH 10). Calcium forms a colourless EDTA complex and frees an equiv. amount of naphthylhydroxamate ion, which imparts a yellow to brick-red colour to the soln. according to the amount present. The extinction is measured in a spectrophotometer at 410 m$\mu$ or in a filter photometer with a blue filter.

W. J. WRIGHT

**3579. The substitution of naphthol green B by other dyes in the preparation of indicator for the complexometric determination of calcium.** J. Pigłowski (Main Lab., Inst. of Glass and Ceramics, Warsaw). *Chem. Anal., Warsaw*, 1956, **1** (4), 331–332.—The end-point given by murexide in the titration of Ca with EDTA is much improved when a green dye, such as naphthol green B, is present. Similar improvement of the end-point is obtained by using the following mixture of reagents—murexide (1·00 g), naphthalene green (0·3040 g), naphthol yellow S (0·6955 g) and sodium chloride (100 g).

K. F. SPOREK

**3580. Chelatometric determination of calcium, magnesium and sulphate in alkali chlorides and brines.** Z. Hošťálek and M. Pollertová (High School Chem. Technol., Prague, Czechoslovakia). *Chem. Průmysl*, 1956, **6** (7), 271–274.—This method, developed for the rapid control of industrial brine and alkali chloride, is based on a known chelato-metric method used in water analysis. Results of the chelatometric determination are very precise down to the following limits—Ca, 1 mg; Mg, 1 mg; and $Na_2SO_4$, 70 mg per litre.

J. BÖSWART

**3581. Spectrographic method for the determination of strontium in minerals.** B. Tuchołka-Szmeja (Inst. of Inorganic Chemistry, Gliwice, Poland). *Chem. Anal., Warsaw*, 1956, **1** (4), 255–262.— Owing to the difficulties in employing chemical methods for the analysis of mixtures of alkaline earths a modification was adopted of the spectrographic method devised by Ells (*J. Opt. Soc. Amer.*, 1941, **31**, 534) in which air - acetylene flame excitation is used. The sample, containing between 0·04 and 0·085 g of Sr, is fused with fusion mixture in a platinum crucible; the melt is crushed, leached with water and filtered. The residue is dissolved in HCl, the soln. is treated with aq. $NH_3$ to precipitate Fe and Al, and filtered. In the filtrate, Sr, Ca and Ba are pptd. as oxalates, the ppt. is ignited at 500°, and the residue is dissolved in HCl. The soln. is boiled, diluted to 100 ml, and introduced into the air - acetylene burner, and the analytical line 4607·3 A is recorded photographically. With samples containing $\not< 0·2$ g of Ca per 100 ml a correction was required owing to the quenching effect on the emission due to Sr. The results showed that 0·25 $N$ HCl had no effect on the values obtained, but that 0·5 $N$ HCl lowered the values due to Sr by 9%; the presence of 0·05 g of Mg per 100 ml lowered the results by 0·85%, and that of 0·1 g of Mg per 100 ml by 14·6%; Ba in concn. up to 0·1 g per 100 ml had no effect on the results.

K. F. SPOREK

**3582. Separation of alkaline-earth elements. III. Electrolytic separation of a small amount of barium in lead and its polarographic determination.** Mutsuaki Shinagawa, Toshifumi Murata and Sadao Oishi (Chem. Dept., Fac. of Sci., Univ. Hiroshima). *Japan Analyst*, 1957, **6** (2), 91–94.—Lead ($< 2$ g) quant. deposits on a platinum cathode from weakly acid soln. (pH 4 to 5, 200 ml) containing 0·1 to 0·5 $M$ Ca acetate or $NH_4NO_3$ and a small amount of Ba ($< 50$ mg), with constant-current (3 amp.) electrolysis, within 30 min. Barium in the Ca acetate soln. provides a polarographic wave ($E_{\frac{1}{2}}$ = $-1·9$ V $vs.$ the S.C.E.) at pH 9 to 11, the height being proportional to its concn. for $< 10^{-4}$ $M$. The presence of $CO_3{}^{2-}$ or $NO_3{}^-$ interferes with the polarography. Carbonate must be removed with $Ca(OH)_2$ and nitrate is removed by evaporation of the electrolyte; the residue is dissolved in HCl and mixed with Ca acetate as supporting electrolyte.

K. SAITO

**3583. Extraction of certain intramolecular compounds by the radioactive indicator method.** I. M. Korenman and F. R. Sheyanova. *Zhur. Neorg. Khim.*, 1956, **1** (4), 852–862; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,664.—An investigation has been made of the extraction of the dithizone complexes of Zn and Cd in relation to pH with buffer soln. of different composition; with acetate and acid phthalate buffer soln. the extraction is more complete than with citrate and citrate - phosphate soln. The dilution of the buffer soln. has practically no influence on the degree of extraction. The influence of the buffer soln. is not due to its ionic strength but to the relative stabilities of the dithizonates and other complexes (acetates, etc.) of the metals. The composition of the buffer soln. does not affect the extraction of the dithizonates of Hg and Co. In the determination of the composition of the products being extracted, the isomolecular series method was used, with measurement of the radioactivity of the extract. For the $Hg^{II}$ complex, a max. was found with a ratio of Hg to dithizone

equal to 1 : 2 at pH 4·1 and a ratio of 1 : 1 at pH 9·8. For the system nitrosonaphthol - $Co^{2+}$, the ratios 2 : 1 in alkaline medium and 3 : 1 in acid medium are confirmed.

C. D. KOPKIN

**3584. Co-precipitation with certain complex compounds obtained by the action of organic precipitants.** I. M. Korenman, A. A. Tumanov, Z. I. Glazunova, Z. V. Kraĭnova and M. N. Barȳshnikova. *Zhur. Neorg. Khim.*, 1956, **1** (4), 863–873; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,638.—A study has been made of the co-pptn. of small quantities of Zn, Cd and $Hg^{II}$ with ppt. of $Cu^{II}$ anthranilate (**I**), $Cu^{II}$ 8-hydroxyquinolinate (**II**), and phenazone tetrabromomercurate (**III**). The percentage of Zn, Cd and Hg co-pptd. with **I** increases with increase of pH. The introduction of $Cl^-$ decreases the co-pptn. of Cd with **II**. On raising the temp., the abs. quantity of Cd co-pptd. with **III** decreases, which infers the increased solubility of **III**, since the quantity of co-pptd. Cd per unit mass of ppt. is unchanged. Indication is given of the practical applications of the results for the separation of traces of Cd from soln. of salts of Zn, Cu, etc.

C. D. KOPKIN

**3585. Iodimetric determination of cadmium.** M. V. Darbinyan and A. A. Arutyunyan. *Izv. Akad. Nauk ArmSSR, Fiz.-Matem., Estestv. i Tekh. Nauk*, 1956, **9** (2), 23–29; *Ref. Zhur., Khim.*, 1957, Abstr. No. 8447.—Precipitate the Cd as CdS with thiourea or with $H_2S$, filter, and wash the ppt. To the ppt. and filter-paper add a known vol. of 0·1 $N$ iodine, allow to stand for 10 to 20 min., preferably in the dark, and titrate the excess of iodine with 0·1 $N$ $Na_2S_2O_3$ to starch. In determining 1·5 to 67 mg of Cd, the error is $\not> \pm 3\%$. The method may be used for the determination of Cd in certain complex compounds.

C. D. KOPKIN

**3586. Gravimetric determination of mercury with Reinecke's salt.** M. P. Anan'evskaya and V. I. Petrashen'. *Trudȳ Novocherkas. Politekh. Inst.*, 1956, **41** (55), 11–14; *Ref. Zhur., Khim.*, 1957, Abstr. No. 19,550.—Improvements are made in Mahr's method (*Z. anal. Chem.*, 1936, **104**, 241) for determining $Hg^{II}$ with Reinecke's salt. (*i*) Add HCl to the soln. to $\simeq 0·5$ $N$, heat almost to boiling-point and add a soln. of Reinecke's salt (50 mg of the salt to each 10 mg of Hg). After 2 or 3 min., filter off the ppt. and wash first with 1% HCl and then with water, dry at 105° to 110° and weigh as $Hg[Cr(NH_3)_2(SCN)_4]_2$. (*ii*) Or, ignite the ppt. to $Cr_2O_3$, oxidise the $Cr_2O_3$ to chromate by fusion with $Na_2O_2$, and determine Cr iodimetrically. The max. error is reduced from 1·1 to 0·4%.

C. D. KOPKIN

**3587. Complexometric titrations. (Chelatometry.) XXIX. Specific screening and determination of mercury.** J. Körbl and J. Přibil (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (4), 667–671.—Thiosemicarbazide (**I**) has been found to be suitable for masking Hg, yielding colourless and water-sol. complexes. With the use of **I**, the complexometric determination of Bi, Pb, Cd and Zn can be carried out in the presence of a large excess of Hg. The reaction between **I** and the complex of Hg with EDTA was used for specific indirect determination of Hg and Bi or Hg and Pb in the presence of each other. *Procedure for Bi in the presence of Hg*—To an acid soln. (pH 1 to 2) add 0·1 $M$ **I** (5 to 25 ml), dilute to between

100 and 200 ml with water, add Xylenol orange indicator and titrate with 0·05 $M$ EDTA (disodium salt) (**II**) till the colour changes from red to yellow. When titrating Pb, previous neutralisation with hexamine to a pH of 5 to 6 is necessary. In the same manner the titration of Cd and Zn can be carried out. For the titration of Hg in the presence of Zn, Pb or Cd, the total amount of metals at pH 5 to 6 is first determined, and after the addition of **I** the demasked **II** is titrated with a soln. of Pb or Zn. A similar procedure is suitable for mixtures of Bi and Hg; the total amount is determined in an acid soln. (pH 2) but hexamine must be added before the equiv. point is reached. J. ZÝKA

**3588. Determination of boron in fluoride salts.** W. J. Ross, A. S. Meyer and J. C. White (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 810–814.—The fluoride, containing < 500 μg of B, is treated with $AlCl_3.6H_2O$ (15 g), water (20 ml) and 12 $M$ HCl (10 ml) and, after solution is complete, the soln. is shaken with a mixture of ethanol (30 ml) and ether (60 ml). A 25-ml portion of the org. layer is evaporated in the presence of water (5 ml) to a total bulk of 2 to 4 ml. This residue is diluted to 10 ml and a 2-ml aliquot is treated with 12 $M$ HCl (two drops) and 18 $M$ $H_2SO_4$ (10 ml), followed by 0·093% carminic acid in 18 $M$ $H_2SO_4$ (10 ml). After 45 min. the absorption is measured at 585 mμ with respect to a reagent blank: the concn. of B is derived from a calibration curve which allows for the reproducible fraction of B extracted. The temperature-independent partition coefficient is given as $0·59 \pm 0·02$ between 13° and 28° under the conditions of the determination, and a reproducibility of 3% on a 200-μg content of B is quoted. D. A. PANTONY

**3589. Applications of anion-exchange resins to determination of boron.** J. D. Wolszon, J. R. Hayes and W. H. Hill (Pennsylvania State Univ., University Park, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 829–832.—The possibility of using anion-exchange resins as a means of separating borate has been investigated, and a general procedure for B has been developed and applied to its determination in glass and steel. After a standard method of attack, the soln. of the material in $K_2CO_3$ or HCl is subjected to anion-exchange chromatography (Amberlite IR-45 and Nalcite HCR mixed-bed resin) and the borate is eluted with water. The eluate is made up to standard vol. and the B is then titrated potentiometrically by a standard procedure; allowance is made for a small loss of B during the separation by means of a blank. By this method, interference from $Fe^{3+}$, $PO_4^{3-}$, $AsO_3^{3-}$, $Sn^{4+}$, $MnO_4^{-}$, $Cr_2O_7^{2-}$, $Mo^{VI}$, formate, acetate, oxalate and $F^-$ is avoided. D. A. PANTONY

**3590. Determination of boron in coal products (tar and pitch).** L. Wnękowska (Dept. of Chemistry of Coal, Mining Inst., Katowice). *Chem. Anal.*, *Warsaw*, 1956, **1** (4), 301–310.—The method described is suitable for the routine determination of traces of B in coal and coke by-products, which may contain organoboron compounds, boric acid and oxide, borosilicates, boron nitride and boron carbide. The sample (1 g) is decomposed by boiling in a Kjeldahl flask with a mixture of conc. $H_2SO_4$ (40 ml), $K_2SO_4$ (10 g), Hg (one drop) and $H_3PO_4$ (ten drops), until clear (7 to 10 hr.). The reaction mixture is then diluted, methanol is added and the ester is distilled in the usual way into dil. NaOH. The distillate is evaporated to dryness and B is

determined colorimetrically, with carmine reagent, according to the procedure by Nowicka-Jankowska and Szyszko (*Anal. Abstr.*, 1957, **4**, 3592). Up to 10 μg of B can be determined satisfactorily. K. F. SPOREK

**3591. Determination of trace contents of boron in graphite, coal, coke and carbon black by the method of spectral emission analysis. I.** S. Skalska and S. Held (Dept. of Technical Physics, Inst. of General Chemistry, Warsaw). *Chem. Anal.*, *Warsaw*, 1956, **1** (4), 294–300.—The method described is suitable for the determination of 0·1 to 0·2 p.p.m. of B in graphite, coal, coke and carbon black used in the construction of atomic reactors. The sample (1 g) is thoroughly ground, mixed with $Ca(OH)_2$ (0·2 g) and water (15 ml) and dried on a sand bath. The residue is ground in an agate mortar, transferred to a platinum boat and ignited in the presence of air at 850° for 1 hr. The prepared sample is then excited, in the presence of $Li_2CO_3$ as carrier, in an a.c. arc between copper electrodes. The intensity of the analytical line 2497·7 A is measured. The lower limit of detection was 0·06 μg of B per g of sample and the coefficient of variation was 12%. K. F. SPOREK

**3592. Spectrophotometric determination of boron in reactor materials. I. Determination in uranyl nitrate and in hydrogen peroxide solutions.** T. Nowicka-Jankowska and H. Szyszko (Inst. of Nuclear Research, Polish Acad. Sciences, Warsaw). *Chem. Anal.*, *Warsaw*, 1956, **1** (4), 285–293.—Boron is an undesirable impurity in reactor materials owing to its high absorption of thermal neutrons. In order to determine its concn. in uranyl nitrate and hydrogen peroxide at the level of 0·1 to 0·5 p.p.m., a simple and rapid procedure, with carmine as reagent, was devised. *Procedure*—In a quartz distillation apparatus boil the uranyl nitrate sample (4 g) with methanol (30 ml) that has been previously freed of B by being distilled in the presence of mannitol and NaOH. Collect the distillate in a receiver containing 1% NaOH soln. (5 ml) and evaporate to dryness. To the residue add water (1 ml), conc. HCl (one drop) and conc. $H_2SO_4$ (5 ml), stir with a quartz rod to dissolve the residue, add 5 ml of carmine soln. (0·05% in conc. $H_2SO_4$), mix, set aside for 1·5 hr. and measure the extinction at 620 mμ. The best results were obtained in the range 0·5 to 5 μg of B. The errors were ±5% for hydrogen peroxide and ±10% for uranyl nitrate in the range 0·1 to 0·2 p.p.m. of B. K. F. SPOREK

**3593. Use of complexones in chemical analysis. LII. Detection of boron, germanium, niobium and tantalum with catechol violet.** V. Patrovský (Centr. Geolog. Inst., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (5), 968–969.—In neutral soln. (pH 6 to 7), catechol violet (**I**) yields coloured complexes with Ge, B, Nb and Ta. The interference of many other elements can be avoided by the addition of EDTA (disodium salt) (**II**) and ammonium oxalate. *Procedure*—To a slightly acid soln. of the sample add an excess of a mixture of 0·1 $M$ **II** and a satd. soln. of ammonium oxalate (1:1), and, after addition of Na acetate, one to three drops of **I** soln. (0·1%). A reddish (B), purple (Ge), or blue-violet (Nb, Ta) colour appears. J. ZÝKA

**3594. Determination of boric acid in nickel-plating electrolytes.** K. A. Nakladova. *Zavod. Lab.*, 1957, **23** (5), 544.—The use of phenol red

indicator instead of bromothymol blue is recommended for the determination of boric acid by the procedure of Yurist *et al.* (*Anal. Abstr.*, 1956, **3**, 662).

G. S. SMITH

**3595. Determination of boric acid and sulphate ion in nickel electrolytes by ion exchange.** D. M. Abesgauz and Z. I. Kheĭfets. *Trudÿ Leningr. Tekhnol. Inst. im. Lensoveta*, 1956, (35), 183–185; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,674.—In the determination of $H_3BO_3$ and $SO_4^{2-}$ in nickel electrolytes, interfering cations are removed by ion exchange (*cf.* Zan'ko and Degtyarenko, *Zavod. Lab.*, 1952, **18**, 49; Lazarev, *Ibid.*, 1952, **18**, 51). The H form of cationite SBS or Wofatit KS is used. Dilute 2 or 3 ml of the nickel electrolyte to 50 ml and pass the soln. through the column at a rate of 4 to 5 ml per min., not allowing the soln. to fall below the level of the resin. Wash the column with 150 ml of boiled $CO_2$-free water and collect the eluate and wash liquor in a 300-ml flask. To determine $H_3BO_3$, neutralise the soln. with 0·5 $N$ NaOH to methyl red, add 5 or 10 ml of invert sugar and titrate with 0·1 $N$ NaOH to phenolphthalein (five drops) with repeated additions of sugar. To determine $SO_4^{2-}$, titrate the soln. with 0·1 to 0·2 $N$ NaOH to methyl red or methyl orange; subtracting the $Cl^-$ content from the total acidity gives the content of $SO_4^{2-}$ in the soln.

C. D. KOPKIN

**3596. Organic reagents for colour reactions with aluminium.** V. I. Kuznetsov and D. A. Drapkina. *Trudÿ Vses. Nauch. Inst. Khim. Reaktivov*, 1956, (21), 18–25; *Ref. Zhur., Khim.*, 1956, Abstr. No. 78,434.—The reaction of various organic groups with Al was studied with 17 azo compounds. It is established that, for contrast and sensitivity, the best reagents are those derived from catechol, in particular stilbazo (**I**) and 8-(3:4-dihydroxyphenylazo)-1-naphthol-3:6-disulphonic acid (**II**) which are satisfactorily stable. They may be used for the detection and determination of Al in the presence of a 100-fold excess of Be. The purification and synthesis of **I** is easier and **I** is better than **II** for determining Al in the presence of Be. The limiting dilution for both reagents is 1 in 1 $\times$ $10^8$.

C. D. KOPKIN

**3597. Impurity analysis of aluminium by radioactivation and scintillation spectrometry.** P. Iredale (A.E.R.E., Harwell, England). A.E.R.E. Report, EL/M 96, 1957, 8 pp.—Radioactivation in a flux of thermal neutrons and subsequent analysis, with a two-crystal $\gamma$-ray spectrometer as described by Peirson (*Nature*, 1954, **173**, 990), are employed to detect small amounts of certain elements without chemical separation. It is possible to distinguish between the various substances in two ways: by their $\gamma$-ray spectra and by their half-lives. Together, these methods enabled an element to be detected unambiguously, provided that the active isotope emits $\gamma$-rays, that the cross-section to thermal neutrons is reasonably large, and that the half-lives of the activities produced are not too large or too small, *i.e.*, are within the range 1 hr. to several days. Quantities of Mn, Ga, Cu, Na, Sb, Hf and Sc of the order of $10^{-5}$ to $10^{-6}$ g are easily detected by this means. The results for impurities detected in 99% pure and "super-pure" aluminium are tabulated.

J. M. JACOBS

**3598. Photometric determination of small quantities of beryllium in aluminium alloys.** L. P. Adamovich and B. V. Yutsis. *Ukr. Khim. Zhur.*, 1956, **22** (4), 523–526; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,745.—A method is evolved for the photometric determination of small quantities of Be in aluminium alloys with thoron. Dissolve the sample of alloy (5 g) in HCl, remove the Al by Havens' method (*cf. Z. anorg. Chem.*, 1898, **16**, 15), and the Cu and Zn electrolytically, and dilute to 100 ml. Titrate 5 ml with $N$ HCl soln. to indigo carmine to pH 12·4, and to another 5 ml of the soln. add the vol. of HCl used in the titration and make up to 10 ml with buffer soln. Take 3 ml of this soln., add 4 ml of buffer soln. and 3 ml of thoron soln. (0·0426 g in 100 ml of water) and measure the extinction, with a green filter and a 20-mm cell, with a comparison soln. of 7 ml of buffer soln. and 3 ml of thoron soln. The method has been checked on a "synthetic alloy" with a content of Be of 0·005%. The max. relative error is 17%.

C. D. KOPKIN

**3599. The electrolytic determination of gallium.** H. Terrey and J. Thabit (Royal Coll. Arts and Sciences, Baghdad, Iraq). *J. Chem. Soc.*, 1957, 3064–3066.—The defects of Reichel's method (*Z. anal. Chem.*, 1933, **87**, 321) were overcome by protecting the platinum-gauze electrodes by a prior deposition of copper. A measured vol. of $Ga_2(SO_4)_3$ soln. was mixed with 25 ml of standard $CuSO_4$ soln. (containing $\simeq$ 0·002 g of Cu per ml), and 5 $N$ NaOH (10 ml) and $(NH_4)_2SO_4$ (3 to 5 g) are added. The copper is deposited by applying a potential of 2 V and the current is increased to 4 to 5 amp. to deposit the gallium. The washed and dried electrode is then weighed and the calculated weight of Cu is subtracted from the increase in weight. The error is less than $-$ 0·05%.

N. E.

**3600. The visual colorimetric determination of thallium with methyl violet.** G. G. Shemeleva and V. I. Petrashen'. *Trudÿ Novocherkas. Politekh. Inst.*, 1956, **41** (55), 35–40; *Ref. Zhur., Khim.*, 1957, Abstr. No. 19,562.—The determination of $Tl^{III}$ is based on the reaction of complex halogen anions of $Tl^{III}$ with methyl violet (**I**) to form sparingly sol. compounds of a blue - violet colour, which may be extracted with organic solvents. *Procedure* —Mix 1 or 2 ml of the neutral soln. of $Tl^{III}$, $N$ HCl (0·7 ml) and a 0·02% soln. of **I** (1 ml). Add water to 5 ml and extract with 5 ml of toluene, which does not extract **I**. The quantity of Tl present is found by comparison with a series of standards.

C. D. KOPKIN

**3601. Polarographic determination of traces of thallium in metallic cadmium and lead and in iron - cadmium masse.** Z. Zagórski and O. Kempiński. *Chem. Anal., Warsaw*, 1956, **1** (4), 273–284.—When Cd and Fe and their oxides are used in alkaline accumulators certain impurities present in these metals have an adverse effect on the performance of the accumulator. Since Tl was suspected of being present as an impurity, an analytical method for its determination was devised. *Procedure*— Dissolve 3 g of cadmium sponge in 30 ml of 7 $N$ $HNO_3$, filter, evaporate the soln. to about 10 ml, cool, add 5 ml of bromine water and extract with ether in a continuous extraction apparatus for 5 hr. Evaporate the extract, treat the residue with $HNO_3$ - $H_2SO_4$ and evaporate to dryness. Dissolve the residue in 5 ml of base electrolyte (300 ml of 25% aq. $NH_3$, 214 g of $NH_4Cl$, 300 ml of satd.

aq. SO$_2$ soln., 2200 ml of water) and polarograph ($E_{\frac{1}{2}} = -0.47$ V vs. the S.C.E.). Results obtained with cadmium sponge containing 0·003% of Tl and with cadmium and lead containing 0·01% of Tl gave average relative errors of 8% and 3%, respectively.　　　　　　　　　　　　　K. F. SPOREK

**3602. Spectrographic analysis of rare-earth elements.** P. M. Polyakov and A. K. Rusanov. *Zavod. Lab.*, 1957, **23** (5), 564–569.—Spectrographic methods for the determination of Y and Yb in rare-earth mixtures, impurities in lanthanum, impurities in cerium, and Pr, Nd, Sm and Er in individual rare-earth elements and mixtures are described.　　　　　　　　　　　　G. S. SMITH

**3603. Radiochemical procedure for individual rare earths.** L. R. Bunney, E. C. Freiling, L. D. McIsaac and E. M. Scadden (Chem. Technol. Div., U.S. Naval Defence Lab., San Francisco, Calif., U.S.A.). *Nucleonics*, 1957, **15** (2), 81–83.—After a preliminary separation from other radioactive elements, the rare earths and Y are transferred in HCl to a bed of Dowex-50 (NH$_4^+$ form) at 87°. Selective elution of the elements is then achieved by using lactate buffers of various strengths and pH values.　　　　　　　　　　　　G. J. HUNTER

**3604. Analysis of mixtures of radioactive isotopes by γ-ray measurements. Application of the method to actinium-227, thorium-227 and radium-223.** D. S. Anthony, J. E. Campbell, G. R. Hagee and E. S. Robajdek (Monsanto Chem. Co., Miamisburg, Ohio, U.S.A.). *Radiation Research*, 1956, **4**, 286–293.—A method is presented for the quant. assay of $^{227}$Ac, $^{227}$Th and $^{223}$Ra in various samples without physical or chemical separation. The growth and decay of γ-ray emission is utilised, and ionisation currents are measured in a pressure-type argon-filled (65 lb per sq. in.) ionisation chamber. Measurements are made periodically over a 40-day interval in order to establish the form of the γ-ray emission curve. The coeff. of variation of ion current ranges from 4·3% for 0·07 μC of $^{227}$Ac in the sample to 0·5% for a sample containing 5·3 μC of $^{227}$Ac.　　　　　　　　　CHEM. ABSTR.

**3605. The solution of certain analytical problems in the rare-earth field by radioactivation analysis.** G. I. Born, K. F. Vaïss and M. G. Kobaladze. *Trudy Komiss. Anal. Khim., Akad. Nauk SSSR*, 1956, **7** (10), 104–118; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,059.—A survey is made of the possibility of determining certain rare-earth elements by radioactivation analysis, and it is shown that, with simple subsidiary methods, it is possible to determine them quant. with satisfactory accuracy in a series of mixtures. A radium - beryllium (500 mg of Ra) neutron source and a counting tube with an aluminium window were used. Methods are given for the determination of Eu in samarium, Dy in yttrium earths with or without Gd, Sm in cerium earths free of Eu, and of Gd in yttrium earths containing little Eu.　　　　　　　C. D. KOPKIN

**3606. The potentiometric determination of lanthanum, cerium, praseodymium, neodymium and samarium.** L. Ya. Polyak and F. M. Shemyakin. *Trudy Komiss. Anal. Khim., Akad. Nauk SSSR*, 1956, **7** (10), 276–288; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,061.—From a study of the pptn. reactions of Ce and La with K$_4$Fe(CN)$_6$ and Na oxalate, it is shown that they are hydrolytic pptn.

reactions, and that therefore in the potentiometric titration it is possible to use any type of hydrogen electrode (quinhydrone, antimony, glass). The possibility is indicated of the potentiometric determination of Ce, La, Nd, Pr and Sm with NaOH soln. The influence of elements of the third analytical group and Mg on the potentiometric titration of Ce has been investigated.
　　　　　　　　　　　　C. D. KOPKIN

**3607. Photometric determination of traces of carbon dioxide in gases.** J. Juránek (Inst. Petrol. Res., Brno, Czechoslovakia). *Chem. Listy*, 1957, **51** (4), 716–721.—The continuous measurement of the intensity of the coloration of phenolphthalein during the reaction of a mixture of gases with NaOH soln. was used for the photometric determination of traces of CO$_2$. The reaction is carried out directly in the vessel of the photometer. The method described is suitable for the determination of CO$_2$ in 10$^{-6}$ % v/v when analysing 200 ml of the gas.　　　　　　　　　　　　J. ZÝKA

**3608. A detector-paper for phosgene.** H. F. Liddell (Chem. Defence Expt. Estab., Porton, Wilts., England). *Analyst*, 1957, **82**, 375.—The mixture of diphenylamine and *p*-dimethylamino-benzaldehyde (**I**) used for the detection of phosgene has the disadvantages that the colour change from white to yellow is not easy to see, especially in artificial light, and that it is sensitive to mineral-acid vapours, which simulate phosgene. A mixture of **I** and N-ethyl-N-2-hydroxyethylaniline (**II**) gives a colour change from white to bright blue, and this is neither brought about nor retarded by reasonable concn. of mineral-acid vapour. A detector-paper is prepared by dipping Whatman No. 1 filter-paper in a soln. of 1·68 g of **II**, 0·75 g of **I** and 2·5 ml of diethyl phthalate in 25 ml of ethanol. The paper must be freshly prepared and dry, but the reagent is stable for months in the dark. When 500 ml of air containing 1 μg of phosgene per litre is drawn through such a paper of diameter 0·110 in., an identifiable blue colour appears.　　A. O. JONES

**3609. Determination of thiocyanate or cyanide in the presence of glycine.** J. R. Hudson and J. R. A. Pollock (Brewing Ind. Res. Foundation, Lyttel Hall, Nutfield, nr. Redhill, England). *Analyst*, 1957, **82**, 374.—Application of the method of Aldridge (*Analyst*, 1944, **69**, 262; 1945, **70**, 474) for the determination of CN$^-$ or SCN$^-$ to aq. extracts of barley or malt gave the characteristic red colour formed when CN$^-$ or SCN$^-$ are present. Investigation showed that the reaction was due to the presence of glycine which, in neutral soln., can react with Br to produce small amounts of cyanogen bromide. Separation of the glycine in the determination of CN$^-$ or SCN$^-$ is, however, unnecessary, because the reaction does not occur in strongly acid soln., and accurate results are obtained if the soln. containing CN$^-$ or SCN$^-$ is acidified before the addition of bromine.　　　　　A. O. JONES

**3610. Separation of small amounts of tin from tungsten by adsorption on silica gel.** Z. Řezáč and M. Roubal (Spolek pro chem. a hutní, Ústí nad Labem, Czechoslovakia). *Chem. Listy*, 1957, **51** (5), 884–889.—For the determination of Sn in tungstic acid, dissolve the sample (5 g) in N NaOH (40 ml), on a water bath. Filter off the residue on filter-paper and wash it with hot H$_2$O. Ignite the filter in an iron crucible, fuse the residue with

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NaOH (1 g), dissolve the melt in hot $H_2O$, filter and combine the filtrates. Add 0·1 $M$ EDTA (disodium salt) (10 ml), and neutralise with dil. $H_2SO_4$ (to methyl red). Dilute with $H_2O$ to 200 ml, adjust the content of sulphate to 0·2 $M$ and add aq. $NH_3$ to pH 9. Pass the soln. through the adsorption column of silica gel and adjust the flow (5 ml per min.). Wash with $H_2O$ (200 ml) and elute the adsorbed $Sn(OH)_4$ with conc. HCl (6 ml) and $H_2O$ (70 ml) into a 100-ml flask containing $NH_4Cl$ (21 g). Mix and add gelatin soln. (0·25%) (1 ml), adjust the vol. and register the polarographic wave from $-0·2$ to $-0·8$ V. The second wave can be used for quant. evaluation. The method is suitable for the determination of as little as $10^{-2}$ to $10^{-3}$ % of Sn. The application to tungsten concentrates is also described.                                    J. ZÝKA

**3611. Determination of small amounts of tin in ores by fluorescent and colorimetric methods.** L. B. Ginzburg and É. P. Shkrobot (State Sci. Res. Inst. of Non-ferrous Metals). *Zavod. Lab.*, 1957, **23** (5), 527–533.—The use of 8-hydroxyquinoline, morin and phenylfluorone for fluorescent or colorimetric determination of $Sn^{4+}$ is studied. The most sensitive are the morin fluorescent and the phenylfluorone colorimetric methods. To determine Sn in ores, the sample (0·25 to 0·5 g) is fused with $Na_2O_2$, the aq. soln. of the melt is neutralised with HCl, 30 ml of HBr and 60 ml of dil. $H_2SO_4$ (1 + 1) are added, and the soln. is distilled in a current of $CO_2$; the portion distilling at 100° to 127° is rejected, and a further 15 ml of HBr is added dropwise when the temp. reaches 200°, the temp. being maintained const. An aliquot portion of the distillate is used for the subsequent procedure (*a*) with morin or (*b*) with phenylfluorone. With (*a*), the soln. (2 to 5 ml) in a 1-cm-diameter cylinder is mixed with a few drops of water saturated with $SO_2$ and 1 ml of 0·05% morin soln. and then neutralised to Congo red paper with aq. $NH_3$ and 10% HCl soln. After addition of 0·4 ml of the acid in excess and dilution to 10 ml with water, the intensity of fluorescence obtained with a mercury lamp is compared with that of standard soln. With (*b*), the soln. (5 to 10 ml) is neutralised to pH 5 (Congo red paper) with aq. $NH_3$, then treated with 1·6 ml of dil. $H_2SO_4$ (1 + 1), 2·5 ml of 0·5% gelatin or gum arabic soln. and 5 ml of 0·03% phenylfluorone soln., and diluted to 25 ml; after 30 min. the soln. is examined photometrically, with a 500-m$\mu$ filter. The methods are suitable for determining a few parts of Sn in 10,000 parts of the sample.                                   G. S. SMITH

**3612. EDTA titration of tin in copper-base alloys.** J. Kinnunen and B. Wennerstrand (Outokumpu Oy, Metalworks, Pori, Finland). *Chemist Analyst*, 1957, **46** (2), 34–37.—Both $Sn^{II}$ and $Sn^{IV}$ form stable EDTA complexes in the pH range of 1 to 6. In the 5 to 5·5 range, Sajo's method for the EDTA titration of Al (*cf. Anal. Abstr.*, 1956, **3**, 732) was found to be applicable to Sn. The Sn is complexed in acid soln. with excess of EDTA (disodium salt), which is then back-titrated with a zinc salt soln., with 3:3'-dimethylnaphthidine - ferro-ferricyanide as indicator. In mixed metal systems, the total metal content is thus titrated. The Sn is then displaced with F⁻ and the EDTA freed is titrated. Tin can also be determined at pH 2 by addition of excess of EDTA and back-titration with $Th(NO_3)_4$ soln., with Xylenol orange as indicator.
                                        W. J. WRIGHT

**3613. Spectrographic determination of tin, lead, antimony, bismuth and cadmium in titanium, zirconium, tantalum and niobium.** Sh. G. Melamed and A. M. Saltýkova. *Zavod. Lab.*, 1957, **23** (5), 573–576.—The two-arc method is used for spectrographic analysis of Ti, Zr, Ta and Nb.
                                        G. S. SMITH

**3614. Complexometric titrations. (Chelatometry.) XXVIII. Micro-determination of lead with dithizone as indicator.** S. Kotrlý (Military Tech. Acad., Brno, Czechoslovakia). *Chem. Listy*, 1957, **51** (4), 730.—*Procedure*—Dilute a slightly acid soln. containing 0·05 to 6 mg of Pb in 10 ml with 45% ethanol to $\geqslant$ 50 ml; add 15 to 20 drops of the indicator (a saturated soln. of dithizone in 96% ethanol), neutralise with dil. aq. $NH_3$ when necessary till a red colour appears. Add hexamine buffer soln. (pH 5·0) and titrate with 0·001 $M$ EDTA (disodium salt) (an ammoniacal buffered ethanolic soln. is used) till the colour changes to greenish blue. Magnesium and alkaline earths do not interfere. The interference of heavy metals can be avoided by extracting the Pb with a $CHCl_3$ soln. of dithizone in alkaline medium, in the presence of ammonium citrate and KCN.                               J. ZÝKA

**3615. Determination of lead by amperometric titration with the use of [copper ions as] an indicator.** Yu. I. Usatenko and M. A. Vitkina (Dniepropetrovsk Chem.-Tech. Inst.). *Zavod. Lab.*, 1957, **23** (4), 427–429.—The determination of Pb in electrolytic baths containing sulphamic acid is described. The sample (10 ml) is diluted to 100 ml and an aliquot (2 to 5 ml) containing 15 to 40 mg of Pb is mixed with 25 ml of water and 0·5 to 1 ml of 0·1 $M$ sodium silicate and heated at 70° for 5 min. to coagulate the colloid. The cooled soln. is mixed with 10 ml of 0·1 $M$ $NH_4Cl$ and treated with a slight excess of 0·1 $N$ NaOH, followed by acetic acid to give a pale-pink colour with methyl red indicator. A rotating platinum micro-electrode is inserted, connection is made with a mercury iodide half-cell, a stirrer is operated, and 1 ml of a soln. of a salt of Cu (concn. not stated) is added. The soln. is titrated without applied e.m.f. with an ammonium oxalate soln. Near the equivalence point the diffusion current due to Cu disappears. The titration curve is drawn and a correction made for the Cu present.                                        G. S. SMITH

**3616. Amperometric and potentiometric titration of lead and copper with ethylenediaminetetra-acetic acid.** Nobuyuki Tanaka, Mutsuo Kodama, Minoru Sasaki and Mieko Sugino (Chem. Dept., Fac. of Sci., Tohoku Univ., Sendai). *Japan Analyst*, 1957, **6** (2), 86–91.—Amperometric titration of $Cu^{2+}$ and $Pb^{2+}$ (both 0·0005 to 0·1 $M$) is satisfactorily carried out with EDTA (disodium salt) in aq. soln. of pH 4·2 containing 0·01% gelatin ($-0·12$ V vs. the S.C.E. for Cu and $-0·60$ V for Pb). On the application of potentiometric titration at a const. current, a marked potential jump takes place before the equiv. point is reached. The deviation was compared with the theoretically predicted one (Tanaka *et al., Anal. Chem.*, 1956, **28**, 1555) and it was found that the proposed empirical formula $C = [(i_a - i_r)(V + v)/kV] + (cv/V)$ [$C$ is the molar concn. of Cu or Pb; $c$, the molar concn. of EDTA; $V$, initial vol. (ml) of the sample soln.; $v$, vol. (ml) of the EDTA soln. used until the indicator electrode reaches the given potential; $i_a$, applied current, in $\mu$A; $i_r$, residual current at the given potential, in

$\mu A$; $k$, sensitivity of the electrode, in $\mu A$ per mole per litre] is valid for these ions in concn. $> 0.0005\ M$.

K. SAITO

**3617. Bismuthiol as an analytical reagent. IX. Determination of lead and silver volumetrically.** Anil Kumar Majumdar and Bhu Ratna Singh (Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1957, **156** (4), 265–268 (in English).—The pptn. of Pb and Ag as the bismuthiol II complexes are carried out according to procedures previously described (*Anal. Abstr.*, 1957, **4**, 2548, 2511). The volumetric determination of Pb is accomplished by dissolving the complex in excess of EDTA (disodium salt) at a pH of about 10 and titrating the excess of EDTA against a soln. of Mg or Pb. Similarly, Ag may be determined by dissolving the complex in an excess of KCN soln. and back-titrating the excess against standard $AgNO_3$ soln., with 1 ml of 5% KI soln. as indicator. For amounts of Pb from 5 to 50 mg, results are within about $\pm 0.7\%$. With Ag $< 20$ mg, results are highly satisfactory. With higher amounts of Ag, results are within $+2\%$.

D. F. PHILLIPS

**3618. Chromotropic acid as an analytical reagent. IV. Determination of titanium in the presence of iron and vanadium.** S. Ya. Shnaĭderman. *Izv. Kievsk. Polytekh. Inst.*, 1956, **17**, 197–203; *Ref. Zhur., Khim.*, 1957, Abstr. No. 8365.—Dissolve the cast iron or steel (0.5 g) in $H_2SO_4$ (1 : 3) (20 ml). Towards the end of this process add 1 or 2 ml of conc. $HNO_3$ to decompose carbides and to oxidise iron. Cool and make up to 50 ml. Transfer 1 or 2 ml (according to the content of Ti) to a 50-ml calibrated flask, carefully neutralise with 10% aq. $NH_3$ until only just acid (the cloudiness formed disperses slowly), and add 1 or 2 ml of 3% ascorbic acid soln. Shake, add 1 or 2 ml of 2% chromotropic acid soln. and make up to volume with buffer soln. (pH 2 to 3.5). Measure the intensity of the colour formed. At pH 4.3 to 7.8 the yellow titanium compound possesses a max. optical density of const. magnitude. It is not bleached by NaF at pH 4.7.

C. D. KOPKIN

**3619. Photometric determination of zirconium with morin.** H. Tůma and N. Tietz (Res. Inst. Mater. and Technol., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (4), 722–725.—Zirconium reacts in an acid medium with morin, yielding a yellow coloration, suitable for photometric purposes. The colour is stable in 0.4 to 0.8 $N$ HCl and reaches its maximum intensity after 1 hr. The calibration curve is linear for 0 to 0.5 mg of Zr in 100 ml. *Procedure*—Dissolve the sample in $HClO_4$ and precipitate Fe and Zr (and perhaps small amounts of other elements) with aq. $NH_3$, filter, dissolve the ppt. in HCl and transfer to a 100-ml flask. Reduce the Fe with ascorbic acid, adjust the acidity to 0.5 $N$ HCl and make up to vol. To a 10-ml aliquot add methanol (5 ml) and morin (0.2% soln. in methanol) (1 ml), set aside for 30 to 120 min. (at 20°) and read the extinction at 436 m$\mu$. Make a blank determination. There is no interference from Cr, Ni or Co even in 20-fold excess.

J. ZÝKA

**3620. Determination of thorium in magnesium alloys that contain zirconium.** G. B. Larrabee and R. P. Graham (Burke Chem. Lab., McMaster Univ., Hamilton, Canada). *Z. anal. Chem.*, 1957, **156** (4), 258–265 (in English).—Thorium is pptd. as the tetra-(*m*-nitrobenzoate), which is decomposed, and the resulting *m*-nitrobenzoic acid is determined polarographically. Interfering Zr is previously removed on an anion-exchange resin. *Procedure*—Carefully dissolve 5 g of magnesium alloy in about 70 ml of 12 $M$ HCl. Make the soln. up to 100 ml, and transfer a 5-ml aliquot to an ion-exchange column which has been previously rinsed with 30 ml of 10 $M$ HCl. When the aliquot has seeped through, wash the column with 60 ml of 10 $M$ HCl. Collect the eluate in a 250-ml beaker and add 50 ml of satd. aq. *m*-nitrobenzoic acid soln. and, gradually, 37 ml of 15 $M$ aq. $NH_3$; then allow to cool to room temp. Precipitate thorium tetra-(*m*-nitrobenzoate) (Graham and Larrabee, *Analyst*, 1957, **82**, 415) by adjusting the soln. to pH 2.15 $\pm$ 0.10 with 7.5 $M$ aq. $NH_3$. Coagulate the ppt. by heating nearly to boiling for about 15 min. Filter through Whatman No. 40 paper and wash with hot water. Dissolve the ppt. by means of hot 4 $M$ $HClO_4$ and add 5 ml of 20% aq. tartaric acid soln. Adjust the pH to the methyl red end-point by adding 15 $M$ aq. $NH_3$ from a burette. Add 35 ml of satd. aq. soln. of K H phthalate, 10 drops of 1-naphthol soln. (1% in ethanol) and make up to 100 ml with water. Polarograph a portion of this soln. in a cell which is held in a water bath at 25.0° $\pm$ 0.05° after de-aerating the soln. with pure N. Assess the Th content by comparing the average height of five waves for the reduction step of *m*-nitrobenzoate with a calibration curve prepared as above from soln. of known Th content. If Zr is known to be absent, the ion-exchange step can be omitted and the original sample weight reduced to 2.5 g. The procedures were tested with synthetic alloy soln. containing Mg, 69 to 85%; Zn, 6 to 7%; Ce, 0 to 10%; other rare earths, 2 to 12%; Zr, 0 to 2%; Th, 2 to 4%; Fe, 0.2%; Al, Cu, Pb and Mn, each 0.01%. The precision, based on the overall spread in the wave heights of the five polarograms recorded, is of the order of $\pm 0.01$ to 0.03% Th. Satisfactory agreement between duplicate determinations is also shown.

D. F. PHILLIPS

**3621. Thermolysis of thorium precipitates. Salts of organic acids.** W. W. Wendlandt (Texas Technol. College, Lubbock, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 800–802.—The thermal decomposition of the thorium salts of stearic, pyrogallic, *m*-hydroxybenzoic, *m*-tolyloxyacetic, benzoic, 2:4-dichlorophenoxyacetic, phenylacetic, cinnamic and anthranilic acids and mercaptobenzothiazole was studied on the thermobalance. In general, decomposition occurred at fairly low temp. (below 150°) and the oxide levels were obtained in the temp. range of 450° to 675°. Detailed descriptions of the thermal behaviour of each salt are given.

G. P. COOK

**3622. Selective precipitation of thorium iodate from a tartaric acid - hydrogen peroxide medium. Application to rapid spectrophotometric determination of thorium in silicate rocks and in ores.** F. S. Grimaldi, L. B. Jenkins and M. H. Fletcher (U.S. Geological Survey, Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 848–851.—The ore or rock, containing 2 to 60 $\mu g$ of $Th^{4+}$, is sintered with $Na_2O_2$ and the cake is extracted with water. After acidification with $HNO_3$, a little $Fe(NO_3)_3$ soln. and $H_2O_2$ are added, followed by KOH to basify to a standard excess. The suspension is filtered, and the ppt. is washed and dissolved in $H_2O_2$ and hot $HNO_3$, the soln. being collected under dil. 8-hydroxyquinoline in dil. $HNO_3$, all reagents being of carefully measured vol. and concn. Tartaric acid and $KIO_3$ are added, followed by $Hg(NO_3)_2$ to act as a carrier for $Hg^{2+}$, and the soln. is cooled and filtered. The

residue is dissolved in HCl and pptd. twice more in the same manner, the filtrate finally being heated with $HClO_4$ and the residue taken up in HCl. The $Th^{4+}$ are determined spectrophotometrically. Variables in the determination are discussed, and accuracy and reproducibility data are presented. Separation from large excesses of $Ce^{4+}$, $Zr^{IV}$, $Ti^{IV}$, $Nb^V$, $Ta^V$, $Sc^{3+}$ and $Fe^{3+}$ is claimed.

D. A. PANTONY

**3623. Colorimetric estimation of milligram quantities of inorganic azides.** C. E. Roberson and C. M. Austin (Quality Evaluation Lab., U.S. Naval Ammunition Depot, Crane, Ind., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 854–855.—The sample, containing 0·5 to 4 mg of $N_3^-$, is boiled with 2% NaOH (two drops) and 30% $H_2O_2$ (0·3 ml) in a distilling flask. After decomp. of excess $H_2O_2$, the soln. is cooled, then diluted with water, and an excess of dil. $H_2SO_4$ (0·6 ml) is added. The soln. is boiled and the distillate containing the $HN_3$ is collected under ice-cold $Fe(NO_3)_3$ soln. (4%) (20 ml), which, after distillation is complete, is made up to 50 ml at 25°. The transmittance is measured at 460 m$\mu$, within 60 min. of mixing, against a reagent blank, and the $N_3^-$ concn. is derived from standards which obey the Beer - Lambert law. There is no interference from $SO_3^{2-}$, $S_2O_3^{2-}$ or $S^{2-}$, but $SCN^-$ and $CNO^-$ are not oxidised by the alkaline $H_2O_2$ treatment, and $NO_2^-$ in the $Fe(NO_3)_3$ soln. render the method ineffective. A coeff. of variation of $\pm 3\cdot 9\%$ is quoted. D. A. PANTONY

**3624. Study of acid and reductant functions of hydrazine sulphate by iodate - iodide mixture.** G. S. Deshmukh and M. G. Bapat (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1957, **156** (4), 269–272 (in English).—When treated with an excess of iodate and iodide, hydrazine sulphate was found to liberate two atoms of iodine per molecule; this was determined by thiosulphate or arsenous oxide in borax - boric acid buffer. The action of iodate - iodide mixture on hydrazine sulphate was studied at different stages of neutralisation and decomposition, and iodimetric relationships were confirmed. Hydrazine sulphate is found to be a satisfactory standard for the titration of iodine in the presence of an alkaline buffer. D. F. PHILLIPS

**3625. Colorimetric determination of phosphorus and silicon as the reduced heteropolymolybdates.** A. K. Babko and L. M. Evtushenko (Inst. of Gen. and Inorg. Chem., Acad. Sci., Ukr.S.S.R.). *Zavod. Lab.*, 1957, **23** (4), 423–427.—The effects of acidity and the reducing agent ($SnCl_2$ or $FeSO_4$) on the colorimetric determination of P and Si by the molybdenum blue method are studied. The lower limit of acidity is determined by the acidity at which free molybdate is reduced, but depends on the nature of the reducing agent. With $SnCl_2$ the concn. of $H_2SO_4$ must be $>0\cdot 8\ N$, and with $FeSO_4$ the concn. must be $>0\cdot 25\ N$. The upper limit of acidity is determined by the decomposition of the heteropoly acid and depends on the nature of the central ion. With the molybdophosphate complex the colour intensity has already started to weaken at $0\cdot 9\ N$ $H_2SO_4$, independent of the nature of the reducing agent. With the molybdosilicate the acidity can be $>5\ N$. In determining P in the presence of Si, the acidity before addition of molybdate should be $>0\cdot 4\ N$. In determining Si, the acidity before addition of molybdate should be $<0\cdot 16\ N$, and during the reduction should be $>1\cdot 6\ N$.

G. S. SMITH

**3626. Volumetric determination of phosphates and of metals in the presence of phosphates. I. Volumetric determination of orthophosphates with bismuth oxyperchlorate solution.** J. A. R. Genge and J. E. Salmon (Battersea Polytech., London). *Lab. Practice*, 1957, **6** (6), 325–326.—An aq. soln. of the phosphate, heated to $\simeq 50°$, is titrated with bismuth oxyperchlorate soln., with thiourea as indicator. A large excess of the indicator, or excessive acidity of the soln., gives a false end-point. Insol. phosphates are dissolved in the min. vol. of $HNO_3$ or 40% $HClO_4$, and diluted with water. Sulphate and chloride affect the end-point. Interference by cations largely depends on their valencies. The procedure is suitable for determining phosphate in the presence of most uni- and bi-valent cations, except $Ca^{2+}$ and $Cd^{2+}$, which can be removed by ion exchange, and $Tl^+$, $Pb^{2+}$ and $Hg^{2+}$.

W. J. WRIGHT

**3627. Infra-red absorption spectra of various phosphorus compunds. I. Alkaline orthophosphates.** A. Mutschin and K. Maennchen (Firma Zschimmer & Schwarz, Oberlahnstein, Germany). *Z. anal. Chem.*, 1957, **156** (4), 241–248.—Infra-red absorption spectrograms of orthophosphoric acid and its sodium salts were taken and compared with those given in the literature. In the range 1 to 15 $\mu$, the absorption bands of orthophosphoric acid and its primary sodium salts are very broad and flat, whereas the bands of the secondary and tertiary sodium salts stand out prominently. In the 15 to 25-$\mu$ region it is possible to detect, for example, 1·3 mg of phosphate in 270 mg of KBr, or 0·5% of phosphate in KBr pellets. In this latter range it is also possible to evaluate quant. the bands of the secondary sodium salts. D. F. PHILLIPS

**3628. Quantitative analysis of phosphoric acid. VII. Concentration of trace amounts of phosphate in a dilute solution or in sea water by co-precipitation with magnesium hydroxide.** Masayoshi Ishibashi and Masayuki Tabushi (Chem. Dept., Fac. of Sci., Kyoto Univ., Sakyo-ku, Kyoto). *Japan Analyst*, 1957, **6** (1), 7–10.—Pptn. of a trace amount of P (5 to 25 $\mu$g) with $Mg^{2+}$ was examined with radioactive $^{32}P$. The pptn. is quantitative when the pptd. Mg is $>7$ mg per 100 ml of the sample soln., irrespective of the excess of $Mg^{2+}$ remaining in the soln. No appreciable co-pptn. of $Cl^-$ or $SO_4^{2-}$ was observed for sea water. The sea water is made basic with $N$ aq. $NH_3$ soln., then centrifuged, and the ppt. is dissolved in a small amount of $HNO_3$ and then determined colorimetrically with ammonium molybdate and $SnCl_2$. Arsenic is similarly pptd. with Mg, but does not interfere with the colorimetry provided that the molar ratio to P is $<3$. The influence of organic P was also examined with Ca glycerophosphate ($<250$ mg), and was not appreciable for $>20\ \mu$g of inorganic P.

K. SAITO

**3629. Photometric determination of arsenic and phosphorus in copper-base alloys.** H. C. Baghurst and V. J. Norman (Defence Standards Lab., Woodville, S. Australia). *Anal. Chem.*, 1957, **29** (5), 778–782.—The copper-base alloy (1·5 g) is dissolved in $HNO_3$ (10 ml) and, after expulsion of nitrous fumes and excess of acid by evaporation, the soln. is made up to 100 ml. Aliquots of 20 ml are made up to 100 ml with, respectively, (a) $4\ N\ HNO_3$ (40 ml), 4·8% sodium molybdate - 3·6% sodium vanadate reagent (20 ml) and water; (b) $2\ N\ HNO_3$ (10 ml), the colour reagent (25 ml) and water; (c) (sample

blank) $2 N HNO_3$ (10 ml) and water. After 15 min. the absorption (4-cm ceils) of soln. (a) is measured with a Kodak 543 filter, and that of (b) in 2-cm cells at constant temp. between 17° and 30°, all with respect to a reagent blank and with allowance for the sample blank (c). Phosphorus and As concn. are derived from calibration curves and a given equation. A slightly simpler procedure for the determination of As in P-free alloys is also given. No interference is observed in moderate concn. of $Zn^{2+}$, $Pb^{2+}$, $Ni^{2+}$, $Al^{3+}$, $Fe^{3+}$, $Mn^{3+}$, $Cd^{2+}$, $Bi^{3+}$ and $Sb^{3+}$, and low concn. of $Sn^{4+}$. Accuracy data are presented for alloys containing up to 0·6% of As and 0·1% of P.  D. A. PANTONY

### 3630. The use of basic diaminotriphenylmethane dyes for the detection of minimal quantities of antimony, gold and thallium. L. N. Lapin and V. O. Geïn. *Trudȳ Komiss. Anal. Khim., Akad. Nauk SSSR*, 1956, **7** (10), 217–222; *Ref. Zhur., Khim.*, 1957, Abstr. No. 11,982.—To reduce the influence of excess of HCl and $NaNO_2$ on the sensitivity of the detection of Sb as $SbCl_6^-$ with di- and tri-phenyl-methane dyes, it is recommended that the complex formed be dissolved in organic solvents. The compound of crystal violet with $SbCl_6^-$ is easily dissolved in benzene or its homologues, colouring them blue - violet. On using diaminotriphenyl-methane derivatives (malachite green or brilliant green), more reliable results are obtained. To 1 ml of the soln. containing Sb as $Sb^{3+}$, add $5 N$ HCl (3 ml), 5% $NaNO_2$ soln. (two drops) and 0·5% ethanolic brilliant green soln. (diluted with water in the ratio 1:3) (0·2 ml). Add 1 ml of toluene to the orange - yellow soln. formed. In the presence of Sb the toluene is coloured green, the intensity of which increases with the concn. of Sb. The detect-able minimum is 0·05 $\mu$g of Sb. An analogous reaction is given by brilliant green with $AuCl_3$ and $TlCl_3$, which react as $AuCl_4^-$ and $TlCl_4^-$. The detectable minima are 0·5 $\mu$g of Au and 2 $\mu$g of Tl. A method is described for the detection of Au in the presence of Tl.  C. D. KOPKIN

### 3631. Micro-analysis with the aid of ion-exchange resins. IX. Detection of small quantities of bismuth with thiourea. Masatoshi Fujimoto (Chem. Inst., Fac. of Science, Tokyo Univ., Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (1), 83–87 (in German).—The orange or orange - yellow colour given by thiourea with aq. soln. of bismuth salts is greatly intensified by adsorption on a cation-exchange resin (e.g., "2·5% DVB"). For max. sensitivity place a few grains of resin on a white spot-plate, add one drop of sample soln., set aside for at least 10 min., add one drop of a satd. soln. of thiourea and examine with a lens ($\times 20$) after about 10 min. The concn. of neutral salts in the sample should not exceed 0·5 $M$; $V^V$, $Cr^{VI}$ and $U^{VI}$ have a marked effect only when present in large quantity. With some ions, comparison with a blank is necessary. The limit of detection is 0·1 $\mu$g in a limiting concn. of 1 in $4 \times 10^5$.  G. BURGER

### 3632. Oxidimetric titration with potassium ferricyanide solution. Determination of vanadium. Mitsuo Kiboku (Fac. of Engng, Kinki Univ., Fuse, Osaka). *Japan Analyst*, 1957, **6** (1), 11–12.—Quadrivalent V ($< 100$ mg) is quantitatively oxidised to $V^V$ with $K_3Fe(CN)_6$ at pH 10·6 to 13·0 in the presence of $Na_5P_3O_{10}$ (I) or $Na_4P_2O_7$ (II) ($\simeq 2$ g) and $Na_2CO_3$. The colour changes from brown through green to blue, and then to colourless near the end-point. An alkaline soln. of $V^{IV}$ is gradually

oxidised by atmospheric oxygen in the presence of I or II. Quinquevalent V is quant. reduced with $K_4Fe(CN)_6$ in $2 N H_2SO_4$ in the presence of I or II ($\simeq 2$ g).  K. SAITO

### 3633. Analysis of mixtures of vanadium oxides. P. P. Makhovka (Belotserkov Agric. Inst.). *Zavod. Lab.*, 1957, **23** (5), 533–535.—The sample (0·2 g) of mixed oxides $V_2O_3$, $VO_2$ and $V_2O_5$, such as occurs with used catalysts in phthalic anhydride production, is heated for 20 to 30 min. with 20 ml of 10% NaOH soln. to give a yellow soln. (due to $V_2O_5$) and black insol. $V_2O_3$. Hot water (50 ml) is added, the soln. is filtered and the residue washed with hot water. The insol. matter is washed into the original vessel, and dissolved in 30 ml of dil. $H_2SO_4$ (1 + 2), and the hot diluted soln. (100 ml) is treated with 0·1 $N$ $KMnO_4$ to oxidise $V_2O_3$ to $V_2O_5$. After complete dissolution of the insol. matter the soln. is diluted to 250 ml, 60 ml of cold dil. $H_2SO_4$ (1 + 2) and four or five drops of 0·2% phenylanthranilic acid in 0·2% $Na_2CO_3$ soln. are added, and the soln. is titrated with 0·1 $N$ ferrous soln. The alkaline filtrate containing the $VO_2$ and $V_2O_5$ is evaporated to 100 ml and treated with excess (10 ml) of dil. $H_2SO_4$ (1 + 1) over that required for neutralisation, followed by addition to the hot soln. of 0·1 $N$ $KMnO_4$ to give a pink coloration. After addition of water and dil. $H_2SO_4$ the soln. is titrated as described above. The result is calculated as $V_2O_5$, representing the sum of $VO_2$ and $V_2O_3$. A second sample weight (0·2 g) is dissolved in 30 ml of dil. $H_2SO_4$ (1 + 2), the soln. is diluted to 200 ml, 60 ml of dil. $H_2SO_4$ (1 + 2) and a few drops of the indicator are added, and the soln. is titrated as described above. If, however, the $V_2O_3$ predominates, the soln. is titrated with 0·1 $N$ ammonium metavana-date in the presence of a 0·1% soln. of Na diphenyl-aminesulphonate in water. Calculations give the contents of all three components.  G. S. SMITH

### 3634. The photocolorimetric determination of niobium. A. A. Popel' and L. P. Maksimova. *Uch. Zap. Kazansk. Univ.*, 1956, **116** (5), 86–90; *Ref. Zhur., Khim.*, 1957, Abstr. No. 19,572.—The determination of Nb as niobium - phosphomolyb-denum blue (Davȳdova and Vaĭsberg, *Zavod. Lab.*, 1947, **9**, 1038) is improved. To determine Nb in alloy steels, dissolve the sample (0·1 g) in $6 N$ $H_2SO_4$ (8 to 10 ml), and oxidise Fe by adding a few drops of conc. $HNO_3$. To prevent hydrolysis of Nb, add to the soln. 2% HF (5 ml) and warm for 10 min. Transfer the soln. to a 50-ml flask, add $6 N H_2SO_4$ (10 ml) and make up to volume. Transfer an aliquot of the soln. to a 50-ml flask, add sodium phosphate soln. (3%) (1 ml), $6 N H_2SO_4$ (1 to 2 ml), $(NH_4)_2MoO_4$ soln. (2%) (4 ml) and 12 to 20 ml of water. Heat to 30° and allow to stand for 5 min. for the full colour to form. Add $6 N H_2SO_4$ (15 to 20 ml) and, within 20 to 30 sec., a 2% soln. of $SnCl_2$. Make up to volume and measure the extinction. The error is $\geqslant 6\%$. The determination takes 30 to 35 min. No interference is caused by Cr, Ni, Si or Tl if present in amounts not exceeding 350, 280, 140 and 2 to 4 times, respectively, the content of Nb.  C. D. KOPKIN

### 3635. Determination of tantalum in titanium alloys by ultra-violet spectrophotometry. B. M. Dobkina and E. I. Petrova. *Zavod. Lab.*, 1957, **23** (4), 421–422.—An application of the pyrogallol method is described. The sample (0·4 g) of titanium alloy is treated for 1 hr. in the cold with 50 ml of dil. HCl (1 + 4) and 5 ml of 20% $NH_4F$ soln. and then

heated until reaction ceases. After addition of $\simeq 20$ drops of conc. $HNO_3$, the soln. is boiled to remove oxides of N, then cooled and diluted to 100 ml. A portion (50 ml) is heated with 50 ml of conc. HCl, 15 ml of water and 5 to 6 g of $H_3BO_3$, then treated with 100 ml of hot 2% tannic acid, boiled for 10 to 15 min. and set aside overnight. The ppt. is filtered off on paper pulp and washed five or six times with 0·5% tannic acid in 2% HCl. It is then ignited and fused with 3 g of $K_2S_2O_7$ with a few drops of conc. $H_2SO_4$ to give a transparent melt. The cooled melt is dissolved in 25 ml of 5% ammonium oxalate soln. and the soln. is diluted to 50 ml. Further dilution is necessary with contents of Ta >5%. The diluting soln. contains 12·5 g of ammonium oxalate and 15 g of $K_2S_2O_7$ in one litre. An aliquot containing between 25 and 350 $\mu g$ of Ta is treated in the same way as the standards. These are obtained from a soln. containing 0·25 mg of Ta in 1 ml. From 0·5 to 1·5 ml of this soln. is diluted with the diluting soln. to 2 ml, then 1 ml of water followed at once by 5 ml of 8 N HCl and 2 ml of 20% pyrogallol soln. (prepared by dissolving 20 g of pyrogallol in 90 ml of water, 9 ml of conc. HCl and 1 ml of 2 M $SnCl_2$ in conc. HCl, filtering and keeping in the dark for 10 days) are added with intermediate mixing. After 10 to 15 min. the extinction at 325 $m\mu$ is measured. Of the interfering elements, Sb, W, Mo and U are absent in alloys of Ti, and Nb have a complex having an extinction one forty-fifth that of Ta.

G. S. SMITH

**3636. The amperometric determination of total sulphur in certain ores and products of their treatment.** A. V. Gordievskiĭ and É. É. Radnaeva. *Trudy Moskov. Khim.-Tekhnol. Inst.*, 1956, (22), 131–134; *Ref. Zhur., Khim.*, 1957, Abstr. No. 8532. —To determine total sulphur in silicate and carbonate rocks, slate, and zinc concentrates, the amperometric titration of $SO_4^{2-}$ is used. Mix the ground sample (0·5 to 2 g) with 6 to 10 g of a mixture of 2 pt. of ZnO and 1 pt. of $Na_2CO_3$, bake in a muffle-furnace at 800° to 850° for 1·5 hr. and extract the cooled mixture with 75 to 100 ml of water. Heat the soln. to b.p., filter into a 100- or 200-ml flask, wash the ppt. with hot 1% $Na_2CO_3$ soln. and make up to volume. Acidify a 10-ml aliquot with $HNO_3$ to methyl orange, add 10 ml of acetone (to reduce the solubility of $PbSO_4$) and titrate with small portions (0·2 to 0·5 ml) of 0·2 N $Pb(NO_3)_2$ at a potential of $-0·8$ V (dropping mercury electrode). The end-point is found graphically.

C. D. KOPKIN

**3637. A comparison of various methods of removing tervalent iron ions in the determination of sulphur in pyrites.** S. M. Efros. *Trudy Leningr. Tekhnol. Inst. im. Lensoveta*, 1956, (35), 91–101; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,801.—The following methods for the removal of $Fe^{3+}$ are studied—(*i*) pptn. with aq. $NH_3$; (*ii*) reduction with metallic aluminium; (*iii*) reduction with ascorbic acid; (*iv*) passage through an ion-exchange column; and (*v*) pptn. of $BaSO_4$ without removal of $Fe^{3+}$, and subsequent pptn. of $Fe(OH)_3$ and solution in HCl. For the determination of the S content of pyrites, methods (*iii*) and (*iv*), which give higher results than do the other methods, are more accurate.

C. D. KOPKIN

**3638. A new qualitative colour reaction for the sulphide ion.** A. P. Kreshkov and L. P. Senetskaya. *Trudy Moskov. Khim.-Tekhnol. Inst.*, 1956, (22), 108–112; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,111.

—A new colour reaction for the sulphide ion with the complex salt (I) of $Hg^{2+}$ with diphenylcarbazide is proposed. On adding $Na_2S$ to a drop of a suspension of **I**, the ppt. completely dissolves with the formation of a red colour. The minimum pH is 9. The reaction may be carried out on filter-paper by adding a drop of an aq. suspension of **I** and a drop of 0·1 N Alkali and treating the mixture with gaseous $H_2S$. The colour of the spot changes from blue to red. The sensitivity of the reaction is 2·5 $\mu g$, and the limiting dilution is 1 in 12 × $10^3$. The reaction is specific; no interference is caused by $S_2O_3^{2-}$, $SO_3^{2-}$ and other ions. The reaction may be used in colorimetric analysis for the determination of $S^{2-}$.

C. D. KOPKIN

**3639. Determination of sulphur trioxide in flue gases. I.** R. A. Mott and K. Parramore (Brit. Coke Res. Ass., 74 Grosvenor St., London). *J. Inst. Fuel*, 1957, **30**, 123–126.—The method in which the gases are bubbled through aq. *iso*propyl alcohol, which absorbs some of the $SO_3$ and enlarges the mist droplets so that they are retained by a sintered glass filter (*cf.* Flint, *Brit. Abstr. C*, 1948, 307; and Corbett, *Brit. Abstr. C*, 1949, 104; *Brit. Abstr. BI*, 1951, 853), is improved for the subsequent turbidimetric determination of $SO_4^{2-}$ by rapid mixing of the $BaCl_2$ precipitant. This is achieved by adding a solution of $BaCl_2.2H_2O$ in 80% *iso*propyl alcohol slowly (one drop per sec. from a microburette) to the swirling acidified alcoholic solution of $SO_4^{2-}$ at 24° to 25°, after which the mixture is allowed to stand for 7 min. The co-efficient of variation is 2%. Flue gases from a domestic coke-fed boiler contained $\simeq 1·5$ p.p.m. of $SO_3$, a concn. which tended to rise at higher combustion rates.

A. R. PEARSON

**3640. Some improvements in the method of direct titration of sulphates.** G. V. Zavarov (Chernorechensk Chem. Works). *Zavod. Lab.*, 1957, **23** (5), 541–542.—Titrations of $SO_4^{2-}$ with soln. of $BaCl_2$ in the presence of alizarin red S as adsorption indicator are studied. With low contents of $SO_4^{2-}$ (0·1 to 0·2% in salts) the prior addition of a suspension of $BaSO_4$ is recommended, and alizarin red S is used both as an acidity and an adsorption indicator. The soln. (25 ml) containing $\not> 0·3$ to 0·5 g of dissolved salts is mixed with 15 ml of acetone, five drops of 0·2% alizarin red S soln. and 2 ml of a suspension prepared by mixing 1 litre of a soln. containing 8·87 g of $Na_2SO_4$ with 1 litre of a soln. containing 15·25 g of $BaCl_2.2H_2O$, pouring off the clear liquid on the following day, adding 2 litres of water, again pouring off the clear liquid after standing, then adding 2·5 g of Mg acetate and diluting the suspension to 100 ml. Conc. acetic acid is added until the colour becomes yellow, with a pale straw tint, followed by 1 ml in excess, and the soln. is titrated during stirring with $BaCl_2$ soln. (1 ml ≡ 5 mg of $SO_4^{2-}$) until a pink tint appears. An indicator correction is applied.

G. S. SMITH

**3641. Photometric determination of selenium with ascorbic acid.** Chozo Yoshimura (Fac. of Engng, Kinki Univ., Fuse, Osaka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (1), 5–6.—At pH < 3, ascorbic acid reduces $SeO_3^{2-}$ and gives a red colloidal soln. in the presence of gelatin; one mole of $SeO_3^{2-}$ oxidises 4·4 moles of ascorbic acid. For < 5 mg of Se, photometric back-titration with iodine soln. is used. The extinction - concn. diagram is linear for < 6 mg of Se, at 430 $m\mu$. No interference results from Te.

K. SAITO

**3642. Oxidation of selenite by alkaline ferricyanide using osmium tetroxide as a catalyst.** G. S. Deshmukh and M. G. Bapat (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1957, **156** (4), 273–276 (in English).—The remarkable catalytic action of osmium tetroxide previously observed (*Anal. Abstr.*, 1957, **4**, 3307) was applied in the oxidation of selenite to selenate, followed by direct or indirect procedures. The $Fe(CN)_6{}^{3-}$ were either titrated with $SeO_3{}^{2-}$ soln. at 8 to 10% overall alkalinity, or *vice versa*, an amperometric or potentiometric end-point being used. The indirect procedure involved amperometric titration of excess of $Fe(CN)_6{}^{3-}$ with $AsO_2{}^{-}$ at 10 to 15% alkalinity, and the $Fe(CN)_6{}^{4-}$ with ceric sulphate, with o-phenanthroline or an amperometric indicator. The reactions involved are—

$$SeO_3{}^{2-} + 2Fe(CN)_6{}^{3-} + 2OH^- = SeO_4{}^{2-} + 2Fe(CN)_6{}^{4-} + H_2O$$

$$2Fe(CN)_6{}^{3-} + AsO_2{}^{-} + 4OH^- = 2Fe(CN)_6{}^{4-} + AsO_4{}^{3-} + 2H_2O$$

$$Fe(CN)_6{}^{4-} + Ce^{4+} = Fe(CN)_6{}^{3-} + Ce^{3+}.$$
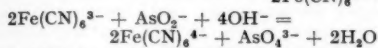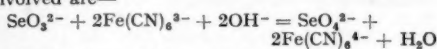
D. F. PHILLIPS

**3643. The visual colorimetric determination of chromium with carmoazine.** N. M. Kravtsova and V. I. Petrashen'. *Trudÿ Novocherkas. Politekh. Inst.*, 1956, **41** (55), 27–34; *Ref. Zhur., Khim.*, 1957, Abstr. No. 19,574.—The determination of Cr as chromate using carmoazine (**I**) [Spenser, *Ind. Eng. Chem.*, 1932, **4** (2), 245] is considerably improved. Mix a 0·1% soln. of **I** (0·4 ml), 5 $N$ $H_2SO_4$ (1 ml), the neutral chromate soln. and water up to 5 ml. Boil for 10 min., cool, and compare the intensity of the blue colour formed with a series of standards prepared analogously. The detectable min. is 0·3 $\mu g$ of Cr in 1 ml. With a content of Cr of up to 3 $\mu g$ per ml, the error is $\not> 11\%$. C. D. KOPKIN

**3644. Determination of impurities in metallic chromium.** N. V. Ageev, A. I. Ponomarev, B. N. Melent'ev and V. A. Trapeznikov. *Zhur. Prikl. Khim.*, 1957, **30** (3), 474–478.—The sample is dissolved in $H_2SO_4$ and $HClO_4$, and Cr is volatilised after addition of HCl. The soln. is evaporated to fuming, $SiO_2$ is separated and determined by the usual method, and the residue after HF treatment is fused with $Na_2CO_3$ or $K_2S_2O_7$, the soln. of the melt in $H_2SO_4$ being added to the main soln. The sesquihydroxides are pptd. by pyridine, the ppt. is fused with $Na_2CO_3$, the melt is extracted with water and the insoluble matter containing the Fe, Ti, Zr and part of the Al is ignited, fused with $K_2S_2O_7$ and dissolved in dil. $H_2SO_4$. Iron is determined by means of 2 : 2'-dipyridyl, Ti by means of $H_2O_2$, both colorimetrically, and Zr is determined gravimetrically by means of phenylarsonic acid. For Al, the ppt. with pyridine is fused with $K_2S_2O_7$, the melt is dissolved in dil. $H_2SO_4$ and Al is pptd. by aq. $NH_3$ after removal of Fe, Ti and Zr by means of cupferron. Manganese is determined volumetrically on a fresh portion of the sample; Ni and Cu are determined polarographically; N is determined by dissolving the sample in HCl and distilling off the $NH_3$; and O is determined by heating the sample *in vacuo* at 850° to give $Cr_2O_3$, followed by dissolution of the metal in dil. HCl and collection of the $Cr_2O_3$.

G. S. SMITH

**3645. Spectrographic determination of very low lead contents in chromium metal and chromic oxide.** J. Niebuhr and C. Pothmann (Forschungsinst. Elchem, Nürnberg). *Arch. Eisenhüttenw.*, 1957, **28** (1), 13–15.—The procedure described enables as little as 0·001% of Pb in chromium to be determined. It avoids time-consuming chemical separation, and reduces the background which limits the sensitivity of procedures that make use of direct volatilisation of the sample in an electrode crater. Metallic chromium is first converted into chromic oxide. A portion (100 mg) is placed in a carbon crucible, which forms the centre of a three-electrode system. Two d.c. arcs with separate circuits are employed, the crucible being the common anode. The lower arc heats the crucible and fractionally volatilises the sample; the upper arc is used, at a lower current value, for spectral measurement. Dimensions, constructional details and experimental conditions are presented. The precision, as coefficient of variation, is $\pm 15\%$ for concn. of Pb of about 0·1%, and $\pm 35\%$ for concn. of Pb. of about 0·001%.

G. BURGER

**3646. The simultaneous determination of chromium, iron and aluminium with EDTA.** R. Patzak and G. Doppler (II Chem. Inst., Univ. Vienna). *Z. anal. Chem.*, 1957, **156** (4), 248–257.—A simple method of complexometric determination depending on variation of pH and temp. is described. Excess of EDTA is back-titrated with $FeCl_3$ soln. Iron is first determined by titration in the pH range 1·5 to 2·0 at a temp. of from 50° to 60°. Iron and Cr are then determined together after previous heating for 5 min. at b.p. and titrating under the same conditions at pH 5·0. This is followed by a similar titration of Fe plus Cr plus Al at pH 5·0 and 40° after 5 min. previous heating at the b.p. Interferences through the presence of other ions, especially Ti, and procedures for preventing these are discussed. D. F. PHILLIPS

**3647. Coulometry of dichromate.** D. Monnier and P. Zwahlen (Univ. Geneva). *Helv. Chim. Acta*, 1956, **39** (7), 1865–1876.—The anodic oxidation of metallic Cr was studied to develop a method of producing, by constant current coulometry, known quantities of $Cr^{6+}$, which can be used for the determination of oxidisable ions by a simple measurement of time. It was found that $Cr^{6+}$ are produced at a chromium anode with a current efficiency which is const. and reproducible for a given anode over a wide range of pH, current density, concn. and number of coulombs used. The anode is standardised permanently by electrolysing a soln. of $N$ $H_2SO_4$ at const. current for a time ($t_1$) and determining the $Cr^{6+}$ by coulometric titration by adding it to an anolyte (5% $K_2SO_4$ soln.) contained in a tube with sintered glass bottom dipping into a catholyte [0·6 $N$ ferrous ammonium sulphate in 4 $N$ $H_2SO_4$ (15 ml), conc. $H_2SO_4$ (1 ml) and conc. $H_3PO_4$ (1 ml)]. The end-point is determined by a redox indicator or potentiometrically with platinum and tungsten electrodes, and the time ($t_2$) is noted. Then current efficiency (%) $= \dfrac{2t_2 \times 100}{t_1}$. This value can then be used for the determination of unknown amounts of $Fe^{2+}$ by coulometric titration in $N$ $H_2SO_4$ with the standardised chromium anode. The precision given is $\pm 0\cdot64\%$ for 10 mg of Fe and $\pm 6\%$ for $\pm 9\cdot5\%$ for two series of determinations on 0·1 mg. G. BURGER

**3648. Improved molybdenum separation procedure.** E. M. Scadden (Chem. Technol. Div., U.S. Radiological Defence Lab., San Francisco, Calif.). *Nucleonics*, 1957, **15** (4), 102.—The classical procedure (NNES IV-9, paper 257) has been

shortened. The first two ppt. of Mo α-benzoin oximate are dissolved in fuming $HNO_3$ and the Mo is re-pptd. directly after partial neutralisation of the $HNO_3$ with aq. $NH_3$. The procedure then closely follows the classical one. The chemical yield is 70 to 75%. Analysis of U eight days after bombardment gave six results having a coeff. of variation of 0·8%. The sources showed the expected decay for 10 days.                                    G. J. HUNTER

**3649. Determination of molybdenum in ore concentrates.** E. V. Ankudimova and V. I. Petrashen'. *Trudȳ Novocherkas. Politekh. Inst.*, 1955, **31**, 73–77; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,072.—The method is based on the reduction of $Mo^{VI}$ to $Mo^V$ in a bismuth reductor in HCl soln., and subsequent titration with an oxidant. After acid decomposition of the concentrate, satisfactory results could not be obtained. Conditions are described for decomposition by fusion with KOH. After fusion, transfer the contents of the crucible to a 250-ml flask. Cool, make up to the mark and, after the ppt. has settled, take two 25-ml aliquots. Add 10 drops of 30% $H_2O_2$ to each, and boil for 1 min. Cool the soln. and neutralise the first aliquot with HCl to phenolphthalein. To the amount of acid used, add the quantity of acid necessary to give an acidity of 1·25 to 1·5 $N$ (6·5 to 8 ml), and add this total amount of acid in one portion to the second aliquot and mix quickly. Discard the first soln. and use the second soln. for the subsequent analysis. As reductor use a 50-ml burette, one-quarter full of metallic bismuth of grain size 0·25 to 0·50 mm. Pass the soln. through the reductor at a rate of 10 to 15 ml per min. Wash out the flask with 10 to 15-ml portions of 1 to 1·25 $N$ HCl and pass the washings through the reductor. Collect the reduced soln. and wash liquors in one flask, add $H_2SO_4$ (sp.gr. 1·84) (12 to 15 ml) and three to five drops of phenylanthranilic acid reagent, and titrate with ammonium vanadate soln. to a red - violet colour. The accuracy of the method is very nearly as good as that of the gravimetric lead molybdate method.        C. D. KOPKIN

**3650. Determination of molybdenum in titanium - molybdenum alloys by means of ion-exchange chromatography.** I. P. Kharlamov and P. Ya. Yakovlev (Sci. Res. Lab., Ministry of Material Stores). *Zavod. Lab.*, 1957, **23** (5), 535–536.—With contents of Mo of 1 to 2%, 0·1 g of sample is dissolved in 50 ml of 10% $H_2SO_4$ soln. and the Mo is separated on $Al_2O_3$. With contents of 2 to 4, 4 to 6 and 6 to 10%, sample weights of 1, 0·5 and 0·25 g are dissolved in 150, 100 and 50 ml, respectively, of 10% $H_2SO_4$, and the Mo is separated on sulpho-coal. The soln. for the separation on $Al_2O_3$ is evaporated to fuming and then diluted to 500 ml. An aliquot (25 ml) is mixed with 0·5 ml of 5% tartaric or citric acid and an excess (one or two drops) of aq. $NH_3$ over that required to neutralise the soln. to Congo red paper is added. The soln. is then allowed to run through a column (1·6 cm in diameter and 4 to 5 cm high) of $Al_2O_3$, followed by 30 ml of 0·5% aq. $NH_3$. The eluate is mixed with conc. HCl (10 ml in excess), 12 to 15 ml of 10% KSCN soln., 5 ml of 10% $SnCl_2$ in 2 $N$ HCl, and water to give 100 ml, and examined in a photometer after 15 min., with a blue filter (400 to 500 m$\mu$). The soln. for the separation on sulpho-coal is diluted so that the content of acid corresponds to between 5 and 6% $H_2SO_4$ and then passed through a column, previously washed with HCl and then water, of sulpho-coal at a rate $\not> 5$ ml per min., followed by water at the same

rate until the washings show no reaction for Ti with $H_2O_2$. The Mo is extracted from the column by means of 150 ml of 5% aq. $NH_3$ and 75 ml of water passed at a rate of 4 to 5 ml per min. The eluate is mixed with conc. $H_2SO_4$ (5 ml in excess of that required to neutralise), then heated to between 80° and 100° and treated dropwise with saturated $KMnO_4$ soln. to give a pink colour. The soln., evaporated to a vol. of 80 to 100 ml and cooled, is passed through a zinc reductor, and the reduced Mo is titrated in an inert atmosphere with 0·05 $N$ $KMnO_4$. No interference is caused by Ti, Fe, Cr, Mn, V, Ni and Co.                    G. S. SMITH

**3651. Spectrophotometric determination of uranium with thiocyanate in butyl Cellosolve - isobutyl methyl ketone - water medium.** O. A. Nietzel and M. A. De Sesa (National Lead Co., Inc., Winchester, Mass., U.S.A.). *Anal. Chem.*, 1957, **27** (5), 756–759.—After the separation of U from interfering ions by extraction into *iso*butyl methyl ketone, with $Al(NO_3)_3$ as a salting agent, the stable colour developed by the extract on the addition of a soln. of $NH_4SCN$ in a 2-butoxyethanol - $H_2O$ solvent is measured at 375 m$\mu$.          E. G. CUMMINS

**3652. Determination of uranium in low-grade ores with hydrogen peroxide.** Hisao Kurama, Yoshihiro Ishihara, Bunshiro Kominami, Toshio Ishikawa and Jiro Ito (Mitsubishi Mining and Metallurg. Lab., Oomiya, Saitama Prefecture). *Japan Analyst*, 1957, **6** (1), 3–6.—A 3 $N$ $HNO_3$ soln. (2 to 4 ml) of phosphate or silicate ore (1 g) containing U ($<0·1\%$) is extracted with ether containing 5% of conc. $HNO_3$ (120 ml) from a cellulose-pulp column (5 g) by the Burstall method (*Analyst*, 1951, **76**, 396; 1952, **77**, 78). In the presence of $PO_4^{3-}$, Fe in an amount greater than that equiv. to the phosphate is added before the elution. The eluate is treated with a mixture of $H_2SO_4$ and $HClO_4$, neutralised and determined colorimetrically with $H_2O_2$ at 420 m$\mu$. The presence of $<10$ mg each of Mo, Cl$^-$, F$^-$ and $PO_4^{3-}$ and $<5$ mg each of Zr, Ti and Ni does not vitiate the estimation; Cr, Mn, Ce, V, Th, Cu and Fe are pptd. as hydroxides under the given conditions.                        K. SAITO

**3653. Chromatographic separation of halogens. I. Chromatography of solutions of halogens in carbon tetrachloride and benzene.** V. B. Aleskovskiĭ and E. S. Boĭchinova. *Trudȳ Leningr. Tekhnol. Inst. im. Lensoveta*, 1956, (35), 102–107; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,847.—Free Cl, Br and I may be separated by passing their soln. in $CCl_4$ through a column of alumina or silica gel. The order of strength of adsorption is $I > Br > Cl$. The primary (Br + I) chromatogram on alumina or silica gel is developed with $CCl_4$ or $C_6H_6$; a clearer Br zone is obtained by treatment with a 1% ethanolic soln. of fluorescein; Cl on silica gel is developed with a mixture of equal vol. of 10% ethanolic soln. of phenol and aniline and 2 $N$ NaOH (giving a green or blue zone). For the qual. analysis of a mixture of Cl, Br and I it is best to use two columns: a silica gel column for Br and a silica gel - starch - silica gel column for Cl and I.
                                       C. D. KOPKIN

**3654. Estimation of fluorine in microgram quantities. A modified procedure.** G. V. L. N. Murty, T. S. Viswanathan and V. Ramakrishna (Tata Iron and Steel Co. Ltd., Jamshedpur, India). *Anal. Chim. Acta*, 1957, **16** (3), 213–215.—When the Willard and Winter method (*Brit. Abstr. A*, 1933,

242) is used in the presence of large excesses of Si, Al or Fe, the recovery of $F^-$ is more rapid and complete if $H_3PO_4$ or $H_2SO_4$ is used instead of $HClO_4$. A soln. of 2 g of ore or slag is treated with 40 ml of the acid and the distillation is conducted at 135° to 140°. The distillate is received in 10 ml of 0·1 N NaOH. To remove $PO_4^{3-}$ and $SO_4^{2-}$, the distillate is first treated dropwise with 1% $AgNO_3$ soln. until no further pptn. occurs; it is then warmed and filtered and the filtrate is treated with 0·1 N $Ba(OH)_2$ in excess and again filtered. The soln. is then neutralised with 0·2 N chloroacetic acid and titrated with standard $Th(NO_3)_4$ (cf. Milton et al., Analyst, 1947, **72**, 43). W. C. JOHNSON

**3655. Determination of the fluorine content of silicates.** Yu. V. Morachevskiĭ and E. N. Egorova (Inst. Chem. Silicates, Acad. Sci., USSR). Izvest. Akad. Nauk SSSR, Otdel. Khim. Nauk, 1956, (11), 1320–1328.—The amount of Si necessary to ensure that the water extract of a soda fusion of a F-containing material contains all the F present is much less than is normally stated. Thus with $CaF_2$ one part of $SiO_2$ with 20 parts of $CaF_2$ is sufficient. In the presence of $Al_2O_3$, however, the content of $SiO_2$ must exceed that of the $Al_2O_3$ to give complete extraction of the F. With the Zr-alizarin colorimetric method for determining F, Si interferes; it is recommended that Si be determined in an aliquot portion of the soln. and that a corresponding amount be added to the standards. For high contents of F, the PbClF method is recommended, but since Si interferes and only small amounts of Al can be tolerated a combination of the PbClF volumetric method, applied to the soln., and the colorimetric method, applied to the ppt., is used. G. S. SMITH

**3656. Spectrographic analysis of silicomanganese.** A. B. Shaevich and M. A. Perepelkina (Lab. of Standard Samples, Ural Inst. of Ferrous Metals). Zavod. Lab., 1957, **23** (5), 556–558.—The powdered sample mixed with graphite and CuO (1 + 20 + 20) is excited in the arc discharge on a moving disc electrode and the lines Si 2532·28, Mn 2533·06 and Fe 2535·60 A are measured. Calibration graphs are constructed with the co-ordinates

$$\left[\Delta S_{\text{Si}-\text{Mn}} \; ; \log \frac{C_{\text{Si}}}{C_{\text{Mn}}}\right] \text{ and } \left[\Delta S_{\text{Fe}-\text{Mn}} \; ; \log \frac{C_{\text{Fe}}}{C_{\text{Mn}}}\right],$$

and the contents of Si ($\simeq 20\%$) and Mn ($\simeq 70\%$) can be determined with an error of 1% of the sample wt. G. S. SMITH

**3657. Spectrophotometric determination of rhenium.** T. R. Andrew and C. H. R. Gentry (Material Res. Lab., Mullard Radio Valve Co. Ltd., Mitcham Junction, Surrey, England). Analyst, 1957, **82**, 372–373.—A soln. of the sample containing 0·5 to 7 mg of Re is mixed with 10 ml of citric acid soln. (20% w/v) and diluted to 100 ml. A 5-ml aliquot is adjusted to a pH of 8 to 9 with 5% NaOH soln., 2 ml of 0·1% w/v aq. tetraphenylarsonium chloride soln. is added, and the liquid is diluted to 10 ml. The liquid is then extracted with 10 ml of $CHCl_3$ and the extinction of the extract (dried over $Na_2SO_4$) is measured at 255 m$\mu$. With tungsten-rhenium and molybdenum-rhenium alloys the sample is fused with NaOH in a silver crucible, and the leached soln. is treated as already described. A. O. JONES

**3658. Polarographic determination of rhenium in solutions.** T. V. Aref'eva, A. A. Pozdnyakova and R. G. Pats. Sb. Nauch. Trudŷ Gos. Nauch. Inst. Tsvet. Met., 1956, (12), 94–98; Ref. Zhur., Khim., 1957, Abstr. No. 15,796.—Polarography of Re with a basal soln. of 4 N HCl and 4 N $HClO_4$ in the presence of 0·005% gelatin gives well-defined waves at 0·25 to 0·30 and $-0\cdot4$ V, the heights of which are directly proportional to the concn. of Re ($\not< 5$ mg of Re per litre). Polarography with a basal soln. of 5 N $H_2SO_4$ gives a well-defined wave at $-0\cdot3$ V for concn. of Re $\not< 1$ to 2 mg per litre, and, with a phosphate buffer basal soln. (pH 7 to 9), a well-defined catalytic wave at $-1\cdot45$ V for concn. of Re of 0·2 to 0·5 mg per litre, the wave height being proportional to concn. in each case. The presence of a 10-fold quantity of Mo and a 4-fold quantity of Cu does not cause interference. There is interference from Ca in the determination of small quantities of Re (5 to 10 mg per litre) with a basal soln. of 4 N HCl and 4 N $HClO_4$ if its concn. in the soln. is >1 to 2 g per litre. C. D. KOPKIN

**3659. The removal of iron in chemical analysis.** S. V. Bleshinskiĭ, V. F. Abramova and A. G. Nagaeva. Trudŷ Inst. Khim., Akad. Nauk KirgSSR, 1956, (7), 19–21; Ref. Zhur., Khim., 1957, Abstr. No. 15,800.—In the removal of Fe by electrolysis with a mercury cathode, instead of a platinum spiral as anode, a lead spiral is used. This is made from lead wire of diam. 5 mm, and is prepared for use by placing it in an electrolyser with 50 ml of water acidified with one drop of $H_2SO_4$ and passing a current for 30 min. The electrolytic separation is carried out in $H_2SO_4$ soln. at 6 to 7 V and 12 amp.; at this current strength, a soln. containing 0·16 g of $Fe_2O_3$ in 70 ml is quant. freed from Fe in 15 min. C. D. KOPKIN

**3660. Spectrophotometric determination of iron with 2-fluorobenzoic acid.** E. B. Buchanan, jun., and W. Wagner (Univ. of Detroit, Mich., U.S.A.). Anal. Chem., 1957, **29** (5), 754–756.—Measurement of the absorption at 525 m$\mu$ of the water-soluble complex formed by the reaction of $Fe^{3+}$ with 2-fluorobenzoic acid is used for the determination of $Fe^{3+}$ in the presence of $Fe^{2+}$. Sixteen interfering ions are listed. E. G. CUMMINS

**3661. Analytical uses of the reaction of bivalent iron with violuric acid.** P. Černý (Inst. Anal. Chem., Komenský Univ., Bratislava, Czechoslovakia). Chem. Listy, 1957, **51** (4), 735–738.—Violuric (5-oximobarbituric) acid (**I**) reacts with ferrous salts, yielding a dark blue soln. at pH 3·9 to 9, with max. extinction at 619 m$\mu$, conforming to the Beer-Lambert law in the concn. range 0·06 to 2·47 $\mu$g of Fe in 1 ml at 665 m$\mu$. By the use of the method of continuous variation, the ratio of 1 atom of Fe to 2 mol. of **I** in the resulting complex was established. The reaction is suitable for the detection of Fe on filter-paper (one drop of 2 N aq. $NH_3$ and one drop of the 1% soln. of **I** in M hydroxylamine hydrochloride), pD = 6·0 Fe. Silver and Hg must be masked with $Na_2S_2O_3$ and KI. Procedure for photometric determination—Measure 10 ml of the sample, containing 0·003 to 0·123 mg of Fe, into a 50-ml flask, add the soln. of M hydroxylamine hydrochloride (1 ml) and after 5 min. add a 1% soln. of **I** (2 ml) and dil. aq. NaOH dropwise till the blue colour appears. Add $NH_4Cl$ soln. (10%) (2 ml) and $Na_2B_4O_7$ (5%) (20 ml), adjust the vol. and measure the extinction at 665 m$\mu$. There is interference from Cu, Co, Zn, Cd, Ag, Hg and $CN^-$. The influence of many other ions is discussed. J. ZÝKA

**3662. Polarographic determination of iron with sucrose as a complex-forming reagent.** O. Manoušek (Res. Inst. Food Technol., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (5), 854–860.—An alkaline soln. of sucrose is a suitable medium for the polarographic determination of Fe. The presence of Mn influences the limiting current of Fe more than in triethanolamine soln. The interference of Mn, Ca and Mg can be avoided by the use of EDTA (disodium salt). *Procedure*—To 3 ml of the soln. of the sample, containing about 1 to $2 \times 10^{-3}$ $M$ Fe, add 2 ml of 0·875 $M$ sucrose, 0·45 $M$ EDTA (disodium salt) (1 to 2 ml), 10 $M$ KOH (1 ml) and dextran soln. (0·1%) (0·5 ml), and adjust the vol. to 10 ml with $H_2O$. Remove O with a stream of N and register the polarographic wave. There is no interference from Ca, Mg or Mn, but Cu and Pb, when present, must first be separated by zinc powder in slightly acid medium. J. ZÝKA

**3663. Phase analysis of iron minerals. I.** Yu. V. Morachevskiĭ and N. Kh. Pinchuk. *Vestnik Leningr. Univ.*, 1956, (10), 60–67; *Ref. Zhur., Khim.*, 1956, Abstr. No. 78,506.—Satisfactory conditions have been found for the separation of magnetite from haematite by using their different rates of dissolution in $H_3PO_4$. Magnetite completely dissolves in a soln. of $H_3PO_4$ and 1% HCl (1:1) by shaking on a boiling-water bath, while haematite is practically insoluble; in the presence of magnetite the solubility of haematite in these conditions is much increased, owing to the presence in the soln. of $Fe^{II}$; on adding an oxidant to the $H_3PO_4$ the solubility of haematite is greatly reduced. The optimum conditions for the separation of magnetite and haematite are a 90-min. treatment at 40° with $H_3PO_4$ (sp.gr. 1·70) and 1% HCl (1:1 by vol.), with the addition of $KMnO_4$ to a pale-pink colour. Under these conditions complete dissolution of the magnetite is accompanied by minimum dissolution of haematite (on the average $\simeq$1% of the total iron in the sample passes into solution). C. D. KOPKIN

**3664. Methods for the analysis of iron and steel. IA. Sulphur in steel.** British Standards Institution (2 Park Street, London). B.S. 1121: Part 1A: 1957, 8 pp.—In this revised method, the sample is dissolved in a strongly oxidising mixture of nitric and hydrochloric acids to convert the sulphur compounds of the steel to sulphate. Nitric acid is subsequently expelled by evaporation and treatment with hydroxylamine hydrochloride, silica is removed by filtration, and the sulphur is pptd. and determined as $BaSO_4$. Slight modification in the procedure to cover alloy steels (other than titanium steel) are suggested.

**XIV. Copper in iron and steel.** British Standards Institution. B.S. 1121: Part 14: 1956, 8 pp.—Two revised methods are described which together cover all classes of iron and steel. Method $A$, based on pptn. as sulphide with $Na_2S_2O_3$, is suited to the determination of low contents of Cu in steels sol. in dil. $H_2SO_4$. Method $B$, based on pptn. as thiocyanate, is applicable to all steels, but less accurate than method $A$ in the low ranges of Cu. In both methods the determination is completed by liberating the iodine equiv. of the Cu with KI and titrating with $Na_2S_2O_3$.

**XXV. Vanadium in iron, steel and ferrovanadium.** British Standards Institution. B.S. 1121: Part 25: 1956, 12 pp.—In this revised method, V is oxidised to $V^V$ with $KMnO_4$ in the cold. Excess of $KMnO_4$ is reduced with $NaNO_2$; sulphamic acid is used to

destroy the excess of nitrite; HF is added and the $V^{5+}$ are titrated with $Fe(NH_4)_2(SO_4)_2$, with pre-oxidised Na diphenylaminesulphonate as indicator.

**XXXVI. Copper in iron and steel (absorptiometric method).** British Standards Institution. B.S. 1121: Part 36: 1956, 8 pp.—Copper in a phosphoric - sulphuric acid soln. is complexed with 2:2′-di-quinolyl and the resulting coloured compound is extracted with amyl alcohol. The content of Cu is determined by measurement of the absorption in the range 540 to 560 m$\mu$ and evaluation from a calibration graph.

**XXXVII. Nickel in iron and steel.** British Standards Institution. B.S. 1121: Part 37: 1957, 11 pp.—The methods described in this Part are of wider application than those described in Part 6: 1948. In method $A$ the sample is dissolved in HCl, then oxidised with $HNO_3$, and the Ni is pptd. as the glyoxime complex from ammoniacal citrate soln. In method $B$, which is applicable to high-cobalt steel, Ni and Co are converted into complex cyanides, the cobalt cyanide is oxidised to the stable cobalti-form with $H_2O_2$, and Ni is pptd. with dimethylglyoxime with the use of formaldehyde to decompose selectively the nickelocyanide. In both methods the filtered glyoxime complex is dissolved in mineral acid and Ni is determined by titration with KCN and $AgNO_3$. J. O. LAY

**3665. The use of elution chromatography from cellulose columns for the systematic analysis of special steels.** G. Venturello and A. M. Ghe (Inst. of Chem. "G. Ciamician", Univ. of Bologna, Italy). *Analyst*, 1957, **82**, 343–352.—The sample is dissolved in HCl according to a specified procedure, and an aliquot ($\equiv$ 10 mg) is applied to the cellulose column previously moistened with acetylacetone. The column is then eluted successively with specified vol. of a series of solvents. Iron and Mo are eluted with acetylacetone; Co and Mn with methyl $n$-propyl ketone containing 5% of conc. HCl; V with a further vol. of the same solvent; Ni and Cr with 8 ml of water and 2 ml of dil. $H_2SO_4$ (1 + 20). The separated metals are then determined absorptiometrically after appropriate colorimetric reactions. Amounts of $\simeq$1 $\mu$g in the eluates can be determined, and the max. error of a single determination is $\pm0·05\%$ for Co, Ni and Cr, and $\pm0·02\%$ for Mn, Mo and V when the results are expressed as a percentage of the sample. A. O. JONES

**3666. New rapid method of carbide analysis [in steel].** A. N. Tananaeva (S. M. Kirov Ural Polytech. Inst.). *Zavod. Lab.*, 1957, **23** (5), 522–527.—The method does not depend on taking a sample of the steel. The steel surface confined by a ring of paraffin wax is attacked electrolytically with Popova's electrolyte (*Zavod. Lab.*, 1945, **11**, 887), but with an increased concn. of citric acid. Results for the separation of the carbide phase are satisfactory only with materials containing stable carbides. G. S. SMITH

**3667. A rapid volumetric determination of vanadium in iron, steel and iron ores.** Shigeo Wakamatsu (Toto Seiko Co. Ltd., Minami-suna-machi, Koto-ku, Tokyo). *Japan Analyst*, 1957, **78** (1), 19–22.—Vanadium is present in the quadrivalent state in an $H_2SO_4$ and $H_3PO_4$ soln. containing both $Fe^{3+}$ and $Fe^{2+}$, and remains unchanged on prolonged ($\simeq$30 min.) heating to white fumes. The presence of Mo and W does not affect the valency state of V, and $H_2O_2$ used for the oxidation of the

sample does not reduce $V^{IV}$ under such conditions. Ammonium persulphate oxidises only $Fe^{2+}$ but not $V^{IV}$. The time taken for an estimation is $<15$ min. The presence of $<5\%$ of Cr does not vitiate the estimation. *Procedure*—(a) Dissolve iron or steel (1 to 2 g) in $H_2SO_4$ (1 + 5, 30 to 50 ml) and conc. $H_3PO_4$ (20 ml) (add $<20$ ml of 30% $H_2O_2$, when necessary) and dilute to $\simeq 400$ ml. Cool to $\simeq 20°$, add $(NH_4)_2Fe(SO_4)_2$ to reduce $V^V$ to $V^{IV}$, then $(NH_4)_2S_2O_8$ (20%, 10 to 15 ml) and titrate with $0.1$ N $KMnO_4$. (b) Dissolve ferrovanadium (0.1 g) in $H_2SO_4$ (1 + 1, 10 ml) and $H_3PO_4$ (10 ml), heat to white fumes of $H_2SO_4$, dilute to 200 ml and treat similarly. (c) Dissolve iron sand (1 to 2 g) in $H_3PO_4$ (30 ml), add $H_2SO_4$ (1 + 1, 10 ml) and treat similarly. K. SAITO

**3668. Photometric determination of titanium in steel by means of chromotropic, gallic and pyrogallolcarboxylic acids.** L. Sommer (Inst. Anal. Chem., Masaryk Univ., Brno, Czechoslovakia). *Chem. Listy*, 1957, **51** (5), 875–879.—A rapid photometric method was developed for steel containing 0·1 to 1% of Ti, even in the presence of 9·8% of Ni and 19·5% of Cr, with the use of chromotropic (**I**), gallic or pyrogallolcarboxylic acid. **I** was found to be the best reagent. The determination must be carried out at pH 3·1 to 3·4 at 470 m$\mu$, and the influence of $Fe^{III}$ is avoided by the addition of ascorbic acid. *Procedure*—Dissolve the sample (0·1 g) in warm $H_2SO_4$ (1:4) (5 ml), filter through a quant. filter into a 100-ml flask, ignite the filter in a platinum crucible and fuse the ash with a small amount of $KHSO_4$. Dissolve the melt in $H_2O$ (5 ml) containing $H_2SO_4$ (1:4) (three drops), filter into the 100-ml flask and make up to vol. Measure 10 ml of this soln. into a 50-ml flask, add ascorbic acid soln. (2%) (10 ml), warm (60° to 70°) and add the soln. of **I** (1% in $H_2O$) (10 ml) and formate buffer soln. (58 ml of conc. formic acid and 28 g of NaOH in 1 litre); adjust to a pH of 3·1 to 3·4 by adding dropwise aq. $NH_3$ (1:1). Cool, and adjust the vol., and after 30 min. measure the extinction (filter S47). Compare with a soln. of the steel (without the reagent) and subtract the extinction of the reagent soln. In the presence of Mo and W, Ti must be first separated as hydroxide after oxidation with $HClO_4$. J. ZÝKA

**3669. Potentiometric determination of ferricyanide with chromium(III) salts.** J. Balej and I. Roušar (Inst. Inorg. Technol., High School of Chem. Technol., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (5), 965–967.—Conditions for a direct potentiometric determination of ferricyanide (**I**) with $Cr^{3+}$ in strongly alkaline soln. are given. *Procedure*—Measure 25·00 ml of 0·05 to 0·1 N **I** into a titration vessel, add 5 N KOH (50 ml), warm on a water bath (50°) and titrate with 0·1 N $KCr(SO_4)_2$ (**II**). The same procedure must be used for the determination of the factor of **II** with pure **I** as standard. The error in 17 determinations did not exceed ±0·8%. The presence of ferrocyanide has no influence on the accuracy of the results. J. ZÝKA

**3670. Estimation of cobalt by benziminazole.** S. P. Ghosh and H. M. Ghose (Dept. Chemistry, Patna Univ.). *J. Indian Chem. Soc.*, 1956, **33** (12), 899–901.—In the method described, Co is pptd. as $Co(C_7H_5N_2)_2$ by aq. benziminazole soln. (1%) at pH 10. The ppt. is dried at 105° and weighed as such. The addition of a little $K_2SO_4$ makes for easier filtration. Between 63 and 12 mg of Co can

be accurately determined in the presence of Ba and Ca. Statistical analysis of the data shows that the method is as efficient as the standard electrolytic and "cobalt sulphate" methods and more easily carried out. O. M. WHITTON

**3671. Separation of cobalt and nickel.** N. S. Fortunatov, V. I. Mikhaïlovskaya and Yu. P. Nazarenko. *Ukr. Khim. Zhur.*, 1956, **22** (4), 536–541; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,805.—The proposed method is based on the pptn. of $Co(OH)_3$ at a lower pH than for $Ni(OH)_3$. Soln. of $Al^{3+}$ or $Cu^{2+}$ salts, in equilibrium with the hydroxides, are used as buffer. To the soln. containing Co and Ni add $Al(OH)_3$ [or $Cu(OH)_2$] and an oxidant (Cl or $HClO_4$) at 70°. The quantity of $Al(OH)_3$ is twice the quantity (in equiv.) of Co, and the oxidant is in slight excess. Separate the ppt. of $Co(OH)_3$ from the soln. (giving filtrate A), wash it with hot water and dissolve it by the action of $SO_2$ to give soln. B. Neutralise soln. B with $Na_2CO_3$ to pH $\simeq 5$, remove the $Al(OH)_3$ and precipitate pure Co by adding $Na_2CO_3$ to pH 7·5. To soln. A, containing Ni and Al (or Cu) add $Na_2CO_3$ to pH 6 and oxidant (Cl); warm for 15 min., set aside for 1 hr. and separate the $Al(OH)_3$. Precipitate pure Ni from the soln. as $NiCO_3$. C. D. KOPKIN

**3672. Polarographic determination of nickel and cobalt ions in absolute ethanol.** Ya. I. Tur'yan, G. É. Teper and L. F. Redchenko. *Trudȳ Komiss. Anal. Khim., Akad. Nauk SSSR*, 1956, **7** (10), 162–169; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,088.—The normal polarographic waves for Ni and Co could not be observed in a 0·1 M $NH_4NO_3$ soln. in abs. ethanol. In 0·5 M $CaCl_2$ soln., Ni gives extended waves, not suitable for quant. measurement, but the wave for Co is better delineated. A direct proportionality between the diffusion current and the concn. of Co was noted. In M $NH_4SCN$ soln. Co does not give a polarographic wave, while Ni gives a very clear wave, the diffusion current being directly proportional to the concn. of Ni. The electro-reduction of the nickel thiocyanate complex is irreversible. With a pyridine basal soln. the waves due to Ni and Co are very clear. In both cases there is direct proportionality between diffusion current and concn. of metal. The electro-reduction of the nickel and cobalt pyridine complexes is reversible. The half-wave potential of Co is 0·22 V lower than that of Ni, which allows the separate waves to be distinguished. The addition of pyridine to a soln. of $CaCl_2$ in ethanol leads to the conversion of the nickel and cobalt chloride complexes into the pyridine complexes. In all the examples investigated, except for 0·1 M $NH_4NO_3$, there are no max. on the polarograms for Ni and Co, although no substances were added as maximum suppressors. C. D. KOPKIN

**3673. Analysis of gases in the chlorination process.** A. S. Berengard and M. T. Glushkova. *Zavod. Lab.*, 1957, **23** (5), 537–539.—The analysis of gases containing volatile metal chlorides, CO, $CO_2$, Cl, $COCl_2$, HCl, O and N, is described. The solid adsorbents Zn and $Sb_2S_3$ are used for HCl and Cl, respectively, iodine in an acetone soln. of NaI is used to absorb $COCl_2$, and normal methods are used for the other components. The gas-analysis apparatus is described. G. S. SMITH

**3674. Rapid routine analysis of free acid, iron, chromium and hydrogen fluoride in hydrofluoric-nitric acid pickle liquor.** Kimio Mukaewaki (Nippon

Tokushu Steel Tube Co. Ltd., Funato, Itabashi-ku, Tokyo). *Japan Analyst*, 1957, **6** (1), 23–26.— Free acid in the pickle liquor is titrated with $N$ NaOH, with a mixture of bromocresol green and methyl red as indicator, $Fe^{3+}$ being masked by the addition of KF (5%, 50 ml for $<10$ ml of the sample). For the determination of Fe, F is removed by heating with $H_3BO_3$ (0·5 g) and HCl (sp. gr. 1·18, 5 ml for 5 to 10 ml of the sample), and $Fe^{3+}$ remaining in the soln. are reduced with $SnCl_2$ and titrated by the Zimmermann - Reinhardt method. Chromium in the sample ($<10$ ml) is oxidised to $Cr_2O_7^{2-}$ by treatment with $H_3BO_3$ (0·5 g) and $HClO_4$ (60%, 10 ml), and titrated with standard $Fe^{2+}$ soln. Fluorine is pptd. as $CaF_2$ with $M$ $CaCl_2$ (10 ml) and $NH_4Cl$ (3 g, to prevent the adsorption of Fe and Cr on $CaF_2$), and the Ca is determined by the usual method.                              K. SAITO

**3675. The systematic analysis of deposits from oil-fired furnaces.** J. W. McCoy (Standard Oil Co., Inc., Richmond, Calif., U.S.A.). *Bull. A.S.T.M.*, 1957, (221), 59–63.—Systematic procedures are described for the determination, in $\simeq 16$ hr., of Fe, Al, V, Pb, Ni, Ca, Mg, Na, Si, S and C in furnace slag and scale after extraction of the sample (10 g) with benzene. Sodium and S are determined on a 1-g portion as sodium zinc uranyl acetate and $BaSO_4$, respectively; C is determined by direct combustion on another portion, whilst the remaining elements are determined in a third portion (0·5 g) after its dissolution in acids and oxidation of C. The acid soln. is evaporated to dryness and the sulphates in the residue are dissolved by treatment with conc. HI, the iodides are oxidised with $HNO_3$, followed by fuming with $HClO_4$. (The acid-insol. residue is usually $<20$ mg and is treated with HF to determine $SiO_2$.) Iron and V are pptd. from HCl soln. as cupferrates, which are then oxidised with $HNO_3$ in $H_2SO_4$ soln. Any $PbSO_4$ is removed and, after reduction to the bivalent state, Fe and V are titrated with $KMnO_4$. Vanadium alone is then determined by the method of Hamner (*Metall. Chem. Engng*, 1917, **17**, 206). Lead is then determined as $PbSO_4$, Al by the urea - basic succinate method, Ni with dimethylglyoxime, and Ca and Mg (in separate aliquots) by the oxalate - $KMnO_4$ and oxine methods. Analyses are of value in the development of anti-scale additives to fuel oil.
                                        W. J. BAKER

**3676. Quantitative analysis of anatase - rutile mixtures with an X-ray diffractometer.** R. A. Spurr and H. Myers (Hughes Res. Lab., Culver City, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 760–762. —Working with the Norelco Geiger-counter X-ray diffractometer, and Bragg angles, from $Cu_{K\alpha}$ radiation, of 12·68° and 13·73° for anatase and rutile, respectively, the ratio $I_A$ to $I_R$ of the intensity of the strongest anatase reflection to the intensity of the strongest rutile reflection has been used as an index of sample composition. The method of calculation is given and results are tabulated.
                                        E. G. CUMMINS

**3677. Spectrographic analysis of dinas and quartzite.** P. F. Lokhov, G. A. Klyushin and M. K. Volkova (Chelyabinsk Metallurg. Works). *Zavod. Lab.*, 1957, **23** (5), 581–584.—The sample in a carbon electrode is excited by an a.c. arc with magnetic stabiliser, and determinations are carried out of MgO, $Fe_2O_3$, $Al_2O_3$, $TiO_2$, CaO and $SiO_2$. The sample powders are mixed with C and $Ba(NO_3)_2$ in the ratio 1:4:1, the Ba line 3071·59 A being used as an internal standard.                      G. S. SMITH

**3678. Auto-radiographic determination of radio-activity in rocks.** A. P. Abrahams (Div. Ind. Hygiene, New York State Dept. Labour, New York). *Nucleonics*, 1957, **15** (3), 85–86.—A method is described for the simultaneous examination of up to 110 samples. Exposures of two to three weeks are used and the concn. of the element is determined from a standard curve. The accuracy of the method is dependent on there being only one type of activity present in a sample.      G. J. HUNTER

See also Abstracts 3520, 3524, 3527, 3528, 3530, 3531, 3534, 3535, 3538, 3543, 3544, 3690, 3721.

# 3.—ORGANIC ANALYSIS

**3679. Recent developments in organic micro-analysis. II. Nitrogen.** G. Kainz (Chem. Lab., Univ. Vienna, Austria). *Öst. ChemZtg*, 1956, **57** (17–18), 242–246.—A review is given of the various suggestions that have been offered for improving the standard methods of Dumas and Kjeldahl. The main difficulty of the former method is to ensure complete oxidation of the compounds, failure to achieve which may lead to a low value of N, some of this being combined with the unburnt C; alternatively, the volume of N may be too large owing to the production of $CH_4$, which is notoriously difficult to oxidise with CuO. The solution offered for these difficulties is to use fine granules of CuO instead of CuO wire, and to heat the furnace to between 800° and 850° instead of at 650°. With the Kjeldahl method, there is difficulty in ensuring complete conversion of N into $NH_3$ because the disappearance of colour from the heated $H_2SO_4$ soln. does not always coincide with the completion of the reaction. Various well-known devices, such as addition of $K_2SO_4$ to raise the b.p. of the acid, or of catalysts such as Hg, and even some org. compounds such as glucose, salicylic acid or phenol, are critically examined and it is concluded that the ideal universally applicable reagent has yet to be found. For liberation of $NH_3$ from the acid mixture, steam-distillation may be replaced by Conway's method of isothermal distillation, particularly for very small quantities.      P. HAAS

**3680. Microchemical determination of sulphur in organic compounds.** W. H. S. Massie (Chem. Dept., Heriot-Watt Coll., Edinburgh, Scotland). *Analyst*, 1957, **82**, 352–358.—The organic compound is oxidised by any standard method yielding $H_2SO_4$, provided that the vol. of the combustion products is not excessive. The soln. of $H_2SO_4$ obtained is neutralised to methyl red with 0·04 $N$ aq. $NH_3$, and is then titrated conductimetrically with 0·1 $N$ Ba acetate soln. The assembly, consisting of a conductivity bridge, test and comparison cells, a micro-burette and a thermostatically controlled oil bath, is described. The accuracy of the method is $\pm 2\%$ on 5 mg of sample.                      A. O. JONES

**3681. Micro-determination of active hydrogen in organic compounds by reaction with lithium aluminium hydride.** A. F. Colson (I.C.I. Ltd., Alkali Div., Winnington, Cheshire, England). *Analyst*, 1957, **82**, 358–362.—Lithium aluminium hydride reacts with H in many types of organic compounds in much the same manner as the Grignard reagents, the vol. of H liberated being a measure of the

active hydrogen content of the sample. An apparatus (illustrated) designed for the micro-determination of unsaturation by hydrogenation (Colson, *Analyst*, 1954, **79**, 298) can be used for this purpose without modification. The solvents used are di-*n*-butyl ether and N-ethylmorpholine, and the purification, storage and dispensing of these solvents and of the LiAlH$_4$ are described. The apparatus includes a device for adding the sample to the mixture of solvent and LiAlH$_4$ and, with the manipulation described, the H evolved is measured over mercury in a gas burette. The method yields results accurate to within $\pm 3 \cdot 0\%$ and reproducible to within $\pm 2 \cdot 5\%$, expressed as coeff. of variation.

A. O. JONES

**3682. Determination of carbonyl groups in oxidised cellulose.** U. Ströle (Zellstofffabrik Waldhof, Mannheim, Germany). *Makromol. Chem.*, 1956, **20**, 19–36.—The methods for the determination of CO groups in oxycellulose (**I**) with NaBH$_4$ are modified in such a way as to decrease the error to 0·01 millimole of CO per g of **I**. **I** is treated *in vacuo* with hydroxylamine, kept at 50° for 2 hr., cooled and brought to atm. pressure; an aliquot is then titrated with 0·1 *N* HCl to pH 3·2. A blank test without **I** is carried out in the same way. The amount $A$ (millimoles of CO per g of **I**) is calculated from $A = [(a \times V/V_a) - (b \times V/V_b)]/10\,g$, in which $g$ is the wt. of **I**, $a$ the ml of 0·1 *N* HCl used in the blank, $b$ the ml of 0·1 *N* HCl used in the main titration, $V$ the total vol., $V_a$ the vol. titrated in the blank, and $V_b$ the vol. titrated in the main experiment. A gasometric method is also described in which the vol. of H evolved in the reaction with NaBH$_4$ is measured. The two methods show good agreement. CHEM. ABSTR.

**3683. Identification of organic compounds. XIII. Use of ion exchangers in organic analysis.** M. Večeřa and F. Friedrich (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (2), 283–286.—The compound to be analysed is converted into a salt, the acid component is liberated on a cation-exchange column and can be isolated or determined titrimetrically. 1-Naphthylmethylthiuronium chloride is suitable for the preparation of the derivatives of fatty acids or sulphonic acids. For disulphonic acids, benzylthiuronium chloride is more suitable, and for amines and thiuronium salts, picrates and 3:5-dinitrobenzoates are recommended. Dialkyl sulphides yield the best derivatives when treated with *p*-bromophenacyl bromide. Water, or a mixture of water with ethanol or acetone, was used as solvent. J. ZÝKA

**3684. Quantitative determination of vinyl chloride and vinyl bromide.** M. Kryszewski and L. Mazur (Inst. Exp. Phys., Univ. Toruń, Poland). *Roczn. Chem.*, 1957, **31** (1), 287–291.—The method is based on the decomposition of vinyl chloride and vinyl bromide in K methoxide soln. at 40° to 50°. The duration of the reaction is 20 min. The KBr or KCl formed is then determined by classical methods. The errors were $\pm 0 \cdot 5\%$ for vinyl bromide and $\pm 0 \cdot 6\%$ for the chloride. The method may be used for the determination of vinyl halides in mixtures with poly(vinyl halides). N. E.

**3685. Colorimetric determination of ethanol by ceric ammonium nitrate.** V. Kratochvíl and C. Soběslavský (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1956,

**6** (12), 515–517.—Determination of ethanol may be made in the range of 0 to 2% in dil. aq. soln., the relative error being $\pm 1\%$. *Procedure*—To the sample (5 ml) add 0·36 *N* Ce(NO$_3$)$_4$.2NH$_4$NO$_3$ in 4 *N* HNO$_3$ (2 ml). Measure the extinction (green filter) and compare with a blank. J. BÖSWART

**3686. Paper chromatography of 3:5-dinitrobenzoates of alcohols.** E. Sundt and M. Winter (Firmenich & Cie., Geneva, Switzerland). *Anal. Chem.*, 1957, **29** (5), 851–852.—The chromatographic separation and identification of the 3:5-dinitrobenzoates of some primary aliphatic alcohols in the range C$_1$ to C$_{12}$ are described. A two-phase system of dimethylformamide - decahydronaphthalene was used for developing, and u.v. irradiation of the chromatogram for detection of the spots.

G. P. COOK

**3687. Improved 2-naphthol and orcinol procedures for differentiation of pentoses and hexoses.** H. Zahnd and S. Sandler (Brooklyn Coll., N.Y., U.S.A.). *Chemist Analyst*, 1957, **46** (2), 39–40.—The colour reactions given by sugars in various procedures are summarised. W. J. WRIGHT

**3688. Rapid detection of reducing substances in paper chromatography.** J. Kellen (Lab. Státních Lázní, Nový Smokovec, Czechoslovakia). *Chem. Listy*, 1957, **51** (5), 973.—For the detection of various reducing sugars in paper chromatography the following reagent has been proposed—a mixture of 0·1 *N* AgNO$_3$, 2 *N* aq. NH$_3$ and 2 *N* NaOH (1:1:2) mixed with an alkaline soln. of KMnO$_4$ (0·5 g of KMnO$_4$ and 1 g of Na$_2$CO$_3$ in 100 ml of H$_2$O). In the presence of reducing sugars a yellow spot, which becomes brown and then black, appears. This will detect 1 $\mu$g of glucose. Formaldehyde and glucosamine give the same reaction. J. ZÝKA

**3689. Determination of acetone in aqueous and benzene solution by Messinger's iodoform method.** G. E. Goltz and D. N. Glew (Univ. of Natal, Durban, S. Africa). *Anal. Chem.*, 1957, **29** (5), 816–818.—The Messinger method for the determination of acetone in soln. is based on the reaction of an alkaline soln. of acetone with an excess of iodine to form CHI$_3$. The excess of iodine is liberated from the alkaline soln. with acid and is determined by titration with Na$_2$S$_2$O$_3$. By this method 102·1 $\pm$ 0·2% of the true acetone content in the range 7 to 41 mg was obtained for aq. soln., and 99·9 $\pm$ 1·1% for benzene soln. The critical factors affecting the determination were studied. G. P. COOK

**3690. The quantitative chromatographic determination of acetate ions in salts of acetic acid.** M. L. Darashkevich. *Trudy Moskov. Khim.-Tekhnol. Inst.*, 1956, (22), 113–115; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,175.—Chromatography is used for the quant. determination of CH$_3$COO$^-$ in the acetates of Na, Mg, Ca, Mn$^{II}$, Co$^{II}$, Zn, Cr$^{III}$, Cd, Cu$^{II}$, Pb and Hg$^{II}$. Dissolve in water that wt. of salt which will give a 0·1 *N* soln. after dilution to 100 or 200 ml. In certain cases (*e.g.*, for cupric acetate) it is necessary to add 5 or 10 ml of 0·1 *N* HCl to prevent hydrolysis. Pass a 25-ml aliquot of the soln. through a column of the cationite SDB–3, wash the column with 100 to 150 ml of CO$_2$-free water, and titrate the eluate and wash liquors with 0·1 *N* alkali to phenolphthalein. C. D. KOPKIN

**3691. Separation, determination and identification of the C₂ to C₆ saturated fatty acids by partition chromatography.** E. Vioque (Inst. de la Grasa, Seville, Spain). *Grasas y Aceites*, 1956, **7** (5), 234–238.—The acids are separated on a silica column, with $H_2O$ as the stationary phase, and a mixture of $CCl_4$ with *n*-butanol (6%) saturated with $H_2O$ as the mobile phase. Bromocresol green may be used to indicate the bands on the column. The acids may be determined by titration with alkali or identified by formation of the *p*-phenylazophenacyl bromide derivatives (purified chromatographically). Recoveries, with amounts of a few milligrams, are usually >90%.            L. A. O'NEILL

**3692. Chromatography of organic acidic compounds on multibuffered paper.** M. Schmall, E. G. Wollish, R. Colarusso, C. W. Keller and E. G. E. Shafer (Hoffman-La Roche Inc., Nutley, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 791–794.— Acidic organic compounds were separated by a paper-chromatographic technique similar to that applied to organic bases (*cf. Anal. Abstr.*, 1957, **4**, 1640). Many acidic compounds form a salt at a particular pH level with alkaline buffer soln. which are applied in sequence of ascending pH on a filter-paper strip in marked zones. A descending chromatographic technique is then used, with $CHCl_3$ as the solvent. Stronger acids are often immobilised at lower pH levels than are the weaker ones; it is thus possible to separate benzoic acid, several barbiturates and some phenolic compounds from each other. These compounds can be made visible in short-wave u.v. light.            G. P. COOK

**3693. Infra-red spectra of aliphatic peroxy acids.** E. R. Stephens, P. L. Hanst and R. C. Doerr (The Franklin Inst., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 776–777.—Peroxypropionic and peroxybutyric acids have been examined in the vapour state from 2 to 15 $\mu$. Infra-red spectra included show absorption at 3·05, 6·9 and 8·5 $\mu$ in addition to C–H and C=O stretching bands.            E. G. CUMMINS

**3694. Volumetric determination of primary amines by nitrite in presence of ferric salts.** M. Matrka and K. Štajner (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1956, **6** (11), 471.—A potentiometric method is used. The sample (1 g) is dissolved in 2·5 $N$ $Na_2CO_3$ (10 ml), and water (60 ml) and conc. HCl (20 ml) are added. The soln. is titrated with 0·5 $N$ $NaNO_2$, with the use of platinum and calomel electrodes.            J. BÖSWART

**3695. Identification of organic compounds. XVI. Identification of sulphides.** J. Gasparič, M. Večeřa and M. Jureček (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (4), 660–666.—From 17 sulphides the corresponding dialkyl-*p*-bromophenacylsulphonium bromides, picrates and perchlorates were prepared and their various physical constants, suitable for identification purposes, were determined. *Procedure*—Boil 0·005 mole of the sample and 0·005 mole of *p*-bromophenacyl bromide in 5 ml of methanol under reflux for 20 min. to 8 hr., according to the character of the reacting compound, and pour into 150 to 200 ml of $H_2O$. The excess of reagent is filtered off. To the soln. of dialkyl-*p*-bromophenacylsulphonium bromide add an aq. soln. of $NaClO_4$ (10%) or a satd. soln. of Na picrate, till a ppt. forms. Crystallise from ethanol or from a mixture of ethanol and ether.

In addition to m.p., the X-ray diffraction data of dialkyl-*p*-bromophenacylsulphonium perchlorates have been found to be the most suitable for identification purposes.            J. ZÝKA

**3696. Determination of thiourea and its methyl derivatives by hypoiodite oxidation, and iodine chloride end-point; iodine bromide end-point in the titration of thiourea with iodate.** G. S. Deshmukh and M. G. Bapat (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1957, **156** (4), 276–280 (in English). —Pre-oxidation by alkali hypoiodite by means of ICl plus KOH or, for thiourea, by iodine bromide, is followed by direct titration with iodate, permanganate or ceric sulphate. The end-point is indicated by the disappearance of the iodine colour from a $CCl_4$ layer, or alternatively by the amperometric dead-stop end-point. In addition to the use of standard oxidants, the methods have the added advantage of a high conversion factor of S to $SO_4{}^{2-}$. A study of factors determining quant. oxidation of thioureas is given. With the dead-stop end-point, as little as 1 ml of 0·0025 $M$ thiourea soln. could be determined with a precision of $\pm1\%$, by using 0·0025 $M$ iodate. The upper limit is set at thiourea concn. equiv. to 25 to 30 ml of 0·025 $M$ iodate. The possibilities of applying a spectrophotometric end-point are discussed.            D. F. PHILLIPS

**3697. Mass spectra of trimethylsilyl derivatives.** A. G. Sharkey, jun., R. A. Friedel and S. H. Langer (U.S. Dept. of the Interior, Region V, Bruceton, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 770–776.— Alcohols, phenols, amines and thiols can be determined by mass-spectroscopic techniques in the presence of other, normally interfering, compounds by the examination of their trimethylsilyl ether derivatives—$ROSi(CH_3)_3$. The appearance of intense rearrangement peaks is discussed and correlation between fragmentation peaks and molecular structure detailed. The mass spectra of 26 aliphatic trimethylsilyl ethers and 8 related silicon compounds are tabulated.            E. G. CUMMINS

**3698. Spectrographic determination of benzene in the main fraction of crude benzene.** S. Z. Shul'ga (Inst. Phys., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1957, **23** (5), 577–579.—Absorption spectra in the u.v. are used for determining the content of benzene. Prior treatment with $AlCl_3$ removes certain interfering impurities.            G. S. SMITH

**3699. New method of analysis for the isomers of dialkylbenzenes.** I. G. Ismailzade, Yu. G. Mamedaliev, Sh. Mirzoeva, T. Zeǐnalova and Kh. M. Abdullaeva. *AzerbSSR Elmler Akad. Kheberlari, Izv. Akad. Nauk AzerbSSR*, 1956, (4), 25–31; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,157.—The existing methods for the separation of dialkyl-substituted benzenes are not accurate. The new method of analysis is based on comparison of the intensities of the characteristic X-ray diffraction lines of the isomers of phthalic acid. Completely satisfactory results are obtained.            C. D. KOPKIN

**3700. Prewaves and postwaves in the polarography of phenyl ketones.** P. J. Elving and J. T. Leone (Univ. of Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 782–784.—Polarographic studies of the effect of irradiation on a soln. of propiophenone in 95% ethanol have shown that the prewave development is due to exposure to u.v. radiant energy. The band is a product of the solvent, but is dependent on the presence of the

ketone. Its development has been prevented by the addition of sodium bisulphite. This addition and the irradiation had no effect on the ketone absorption. The postwave was due to acetaldehyde.
E. G. CUMMINS

**3701. Determination of 4-chloro-2-methylphenoxyacetic acid by the use of its infra-red spectrum.** Shigeyuki Tanaka (Inst. of Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo, Tokyo). *Japan Analyst*, 1957, **6** (1), 33–34.—The determination of 4-chloro-2-methylphenoxyacetic acid (**I**) was studied in the presence of 2-methylphenoxyacetic acid (**II**), 4:6-dichloro-2-methylphenoxyacetic acid (**III**) and 6-chloro-2-methylphenoxyacetic acid (**IV**) in acetone, by the use of i.r. spectra. **IV** is easily removed by distillation. Since the intensity of the key bands for **I** (12·5 $\mu$) is extremely high, the "compensation method" was employed, as follows. The extinction coeff. of a mixture of **I**, **II** and **III** is measured *vs.* **I** soln. of 150 mg per ml. **II** and **III** are determined from their key bands at 13·24 and 13·06 $\mu$, respectively. The working curves are linear for <30 mg of **II** or **III** per ml, the standard deviation being <0·5%. The amount of **I** is calculated from these data by subtraction from 100%.
K. SAITO

**3702. Spot test for *p*-nitroaniline.** F. Feigl and C. Stark-Mayer (Lab. de Producas Mineral, Min. Agric., Rio de Janeiro, Brazil). *Chemist Analyst*, 1957, **46** (2), 37–38.—In a wet process for detecting *p*-nitroaniline in the presence of its isomers, the yellow, aq. ethanolic soln. is treated with *p*-dimethylaminobenzaldehyde and ascorbic acid. No coloured Schiff base is produced in the cold or on warming. If, however, the mixture is evaporated to dryness, the *p*-nitroaniline is reduced by ascorbic acid to the diamine, which forms a red Schiff base. The limit of detection is 0·5 $\mu$g. The sensitivity is reduced if considerable quantities of isomers are present, since their yellow colour tends to hide the red coloration.
W. J. WRIGHT

**3703. The potentiometric titration of hydrazides of carboxylic acids and their condensation products with aldehydes with sodium nitrite.** L. M. Litvinenko, D. G. Arlozorov and V. I. Koroleva (A. M. Gorki Kharkov State Univ.). *Ukr. Khim. Zhur.*, 1956, **22** (4), 527–530; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,924.—An earlier method for the determination of aromatic amines by potentiometric titration with NaNO$_2$ in acid medium in the presence of KBr (*Anal. Abstr.*, 1956, **3**, 1069) is used. Dissolve the sample ($\simeq$0·25 millimole) in glacial acetic acid (15 ml), and make up to 25 ml with conc. HCl. Take a 5-ml aliquot ($\simeq$7 to 15 mg of substance), add $\simeq$0·5 g of solid KBr, insert a platinum electrode and titrate with 0·01 $N$ NaNO$_2$, with vigorous mechanical stirring. The end-point is determined by the max. increase of potential ($\Delta\epsilon/\Delta$V). About 70 hydrazides and their corresponding hydrazones have been analysed by this method. The accuracy is no lower than that of the analysis of aromatic amines, and in several cases is greater than the accuracy of the classical Dumas method for the determination of N.
C. D. KOPKIN

**3704. Paper chromatography of polynuclear aromatics.** T. Wieland and W. Kracht (Inst. für Org. Chem., Univ., Frankfurt a. Main). *Angew. Chem.*, 1957, **69** (5), 172–174.—Ascending chromatography on partly acetylated filter-paper was found to be the most suitable method of separating condensed aromatics. Generally the best separation was obtained with methanol - ether - water (4:4:1). A mixture of methanol and water with either benzene (6:1:2) or light petroleum (20:1:5) was sometimes preferable. The spots were located by u.v. fluorescence (254 m$\mu$). Non-fluorescent materials were detected by their u.v. absorption. Spraying with compounds, *e.g.*, fluorescein or acridine, whose fluorescence is quenched by light-absorbing materials also serves for detection. A table shows ten compounds from C$_{12}$ to C$_{20}$ which were separated, and gives their formulae, number of double bonds, fluorescence in u.v., and $R$ values relative to 3:4-benzopyrene.
G. BURGER

**3705. The system naphthalene - thionaphthen.** S. V. R. Mastrangelo and R. W. Dornte (Allied Chem. & Dye Corp., Glenolden, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 794–797.—The system was investigated by using both the calorimetric and the freezing curve methods to determine the equilibrium regions bounded by the liquidus and solidus curves. Theoretical analysis permitted an accurate prediction of the solid - liquid equilibrium, and the heat of fusion was determined as a function of composition. The data provided the means of determining the thionaphthen content of a naphthalene sample; a value of 1·25 mole-% was obtained as against a value of 1·24 mole-% by spectrophotometric and i.r. methods.
G. P. COOK

**3706. Spectrographic determination of thiophen and carbon disulphide in crude benzene.** O. V. Fialkovskaya (Inst. Phys., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1957, **23** (5), 579–581.—The infra-red spectrum is measured at 7·99 and 4·55 $\mu$ for determining thiophen and carbon disulphide.
G. S. SMITH

**3707. The molecular spectroscopic group-analysis of saturated hydrocarbons.** H. Luther and H. Oelert (Inst. für Chem. Technol., Braunschweig, Germany). *Angew. Chem.*, 1957, **69** (8), 262–266.—The absorption coefficients at some key i.r. frequencies of 5- and 6-membered ring naphthenes, *e.g.*, alkyl-*cyclo*pentanes and -*cyclo*hexanes, supplementary to intensity measurements on i.r. bands of paraffins, have been measured. An analytical method has been established for the determination of long- and short-chain alkyl, methyl and naphthene groups in mixtures of saturated hydrocarbons. The results are compared with values of other structural analyses for hydrocarbon mixtures.
C. A. SLATER

**3708. Analysis of hydrocarbon systems by mass spectra.** K. I. Zimina, A. A. Polyakova and N. S. Sosina. *Zhur. Neorg. Khim.*, 1956, **1** (6), 1264–1270; *Ref. Zhur., Khim.*, 1957, Abstr. No. 12,023.—The results are given of the use of the mass spectrometer in evolving a method for the analysis of mixtures of isomeric hydrocarbons. The mass spectrometer MS-1 has been improved; it has been fitted with automatic dispersion of the mass spectrum, an electronic potentiometer and a system for inserting the sample into the instrument (illustrated). The spectra of the individual hydrocarbons and their 2- and 4-component mixtures are photographed. On the basis of the spectra of the hydrocarbons, coeff. were calculated of the sensitivity and the relative ionisability, these coeff. being necessary for the calculation of the composition of the mixtures. The accuracy of the preparation of the mixtures was 1·5%. The calculation of

the composition of 2-component mixtures was carried out from the sensitivity coeff. and by a relative method, and of 4-component mixtures by the relative method. The reproducibility of the results was within 1%. The mean relative error in the determination of the components was 5·8%.

C. D. KOPKIN

**3709. Potentiometric method for determination of carbonyl sulphide in petroleum gases.** D. B. Bruss, G. E. A. Wyld and E. D. Peters (Shell Devel. Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 807–809.—The sample gas is passed through two scrubbers, the first containing 30% NaOH which removes $H_2S$ and thiols, and the second containing alcoholic monoethanolamine to remove the COS. The contents of the second scrubber are titrated potentiometrically with $AgNO_3$ in acidic alcoholic soln. Concn. as low as 2 mg per cubic meter of gas can be determined, and recoveries >93% were generally obtained. Low recoveries (80%) were found when the scrubber soln. were exposed to sunlight. Results were in good agreement with those from a mass-spectrometric method and a lamp method for total S.        G. P. COOK

**3710. Colorimetric method of determining arsenic in coal and tar.** Z. Flum. *Paliva*, 1957, **37**, 33–36.—The colorimetric method depending on the reaction of Ag diethyldithiocarbamate with arsine is accurate and sensitive and requires fewer precautions than the Gutzeit method. It is simpler and quicker and as accurate as the molybdenum blue method recommended by the I.S.O. A concn. of even 0·0002% can be determined. Reproducibility depends on homogenisation of the sample of tar, on its decomposition and the separation of arsine; it is about 3·3% for coal and 8·0% for tar. The As concn. was 0·0032 to 0·0056% in brown-coal tars and 0·0004 to 0·0036% in brown coals. Non-volatile As was 85 to 92% of total As.        FUEL ABSTR.

**3711. Determination of the concentration of colouring matter.** P. Raymond and E. L. Krugers Dagneaux (N.V.P. de Gruyter en Zoon, 's-Hertogenbosch, Netherlands). *Chem. Weekbl.*, 1957, **53** (12), 134–136.—The concn. of colouring matters (as sold commercially) for food can usually best be determined by titration with $TiCl_3$. The reduction gives a colourless soln. For the preparation of 0·1 $N$ $TiCl_3$, 150 ml of conc. HCl is added to 200 ml of 15% $TiCl_3$ soln., then made up to 2 litres and set aside for two days under an atmosphere of H. The soln. is standardised against $K_2Cr_2O_7$. The sample, equiv. to $\simeq$ 20 ml of 0·1 $N$ $TiCl_3$, is diluted to 200 ml, and is titrated at boiling-point in the presence of 15 g of Na citrate (a) or Na H tartrate (b). Colouring matters permitted in the Netherlands are given in a list, with their mol. wt. and titration factors and an indication whether method (a) or (b) should be used. With mixtures, the ratio of the components can be determined chromatographically and the total by titration. The reproducibility is stated to be good.        G. BURGER

**3712. Determination of vat dyestuffs on the fibre.** L. M. Golomb (Rubezhansk Section, K. E. Voroshilov Sci. Res. Inst. of Organic Intermediates and Dyestuffs). *Zhur. Prikl. Khim.*, 1957, **30** (2), 329–332.—The relative effects of various organic solvents, glycerol, ethanediol, triethanolamine, 2-ethoxyethanol and triethylene glycol, present in a soln. also containing NaOH and dithionite, on the removal of vat dyes from printed calico are

studied. Triethylene glycol is as effective as 2-ethoxyethanol which is normally used for the purpose, and the soln. of the leuco dyes are stable and easily analysed colorimetrically. Polycyclic ketone dyes are easily removed from fibres in the cold, but thioindigo dyes require heat.        G. S. SMITH

**3713. Analysis of synthetic detergents.** J. Ruiz Cruz (Inst. de la Grasa, Seville, Spain). *Grasas y Aceites*, 1956, **7** (5), 243–250.—Qualitative and quantitative methods for the analysis of anionic, cationic and non-ionic detergents, singly and in admixture, are reviewed.        L. A. O'NEILL

**3714. The determination of linalol in essential oils.** Essential Oil Sub-Committee, Analytical Methods Committee, Society for Analytical Chemistry. *Analyst*, 1957, **82**, 325–329.—Of several methods investigated, two were found worthy of more exhaustive consideration. The method of Glichitch (*Bull. Soc. Chim. France*, 1923, **33**, 1284) is satisfactory provided that strict attention is paid to the purity of the reagents, but determination by this method takes four days. The oil (10 ml), dried over anhyd. $Na_2SO_4$ and cooled to 0°, is added to a freshly prepared, cooled aceto-formic acid reagent (prep. described). The mixture is maintained at 20° to 22° for 72 to 96 hr. and, after admixture with 50 ml of ice-cold water, for a further 2 hr. The separated, formylated oil is washed successively with water, $NaHCO_3$ soln. and water, and its ester value is determined. In the more rapid but less dependable method of Fiore (*News Capsule*, 1943, **1**, No. 15), the dried cooled oil is treated with dimethylaniline, acetyl chloride and acetic anhydride. The mixture is cooled for a few min., set aside at room temp. for 30 min. and then maintained at 40° for 30 hr. The acetylated oil is washed according to a specified procedure with $Na_2SO_4$ soln., dil. $H_2SO_4$, aq. $NaHCO_3$ - $Na_2SO_4$ soln. and $Na_2SO_4$ soln., and its ester value is determined.        A. O. JONES

**3715. Use of a polarographic method for control of polymerisation processes.** M. N. Platonova. *Zavod. Lab.*, 1957, **23** (5), 539–540.—Vinyl cyanide is determined polarographically at −2·05 V in 50% ethanol containing 0·01 $M$ tetraethylammonium chloride.        G. S. SMITH

**3716. Determination of traces of *iso*cyanate in urethane-based polymers.** K. A. Kubitz (E.I. du Pont de Nemours & Co. Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 814–816.—Two methods are described. In the quant. method, residual *iso*cyanate is treated with an excess of standard $n$-butylamine in tetrahydrofuran soln. and the excess of reagent is determined colorimetrically by malachite green, with which it forms a colourless derivative. The qual. method depends on the reaction of −NCO groups with the colourless secondary amine derived from malachite green and $n$-butylamine, to form a product with the characteristic colour of malachite green. Recoveries of −NCO from a standard sample ranged from 107 to 115% at the 0·4 to 2·0 × $10^{-6}$ molar level. The qual. procedure will detect less than 0·005 millimole per g of sample.        G. P. COOK

**3717. Determination of monomeric ε-caprolactam and moisture in 6-nylon.** H. H. Schenker, C. C. Casto and P. W. Mullen (E.I. du Pont de Nemours & Co., Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 825–829.—The $H_2O$ and

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$\epsilon$-caprolactam are separated from the sample by vacuum extraction in a specially designed apparatus at 200°, the monomer and part of the $H_2O$ being condensed in a water - ice trap. The monomer is determined by refractive index measurement of an aq. dilution of the condensate, and the $H_2O$ by difference. At the 8% monomer and 1·70% moisture levels the standard deviations were $\pm 0·26$ and $\pm 0·25\%$, respectively, and at the 0·53% monomer and 0·47% moisture levels they were $\pm 0·03$ and $\pm 0·06\%$.                 G. P. Cook

**3718. Qualitative analysis of plastics. I. Identification of cellulose and its derivatives.** E. Biasotto Manó and L. C. O. Cunha Lima. *Rev. Quim. Ind., Rio de Janeiro*, 1956, **25**, 18–24.—A scheme for the identification of cellulose and its derivatives in plastics is given.                     H. Pritchard

**3719. Analysis of [paint and] lacquer media.** S. Høgh (Gladsaxevej 300, Copenhagen). *Färg och Lack*, 1956, **2** (4), 73–98.—Volatile matter is separated by steam-distillation, and inorganic matter by shaking with HCl. The residue is saponified with alcoholic KOH. The alcohol is distilled off, the residue is dissolved in water and the unsaponifiable matter extracted with diethyl ether. The aq. layer is acidified and any phenols are removed by steam-distillation. The resin and fatty acids are extracted with diethyl ether and separated by esterifying the fatty acids with methanol by Wolff and Scholze's method. If phthalates are present, they are separated as $C_6H_4(COOK)_2.C_2H_5OH$ by Kappelmeier's method. With styrenated alkyds, the ppt. is contaminated with polystyrene, which must be removed by extraction with benzene and, after removal of benzene and monomeric styrene by evaporation, washed with 87% methanol before weighing. With nitrocellulose lacquers, an entirely different procedure is followed. The dried lacquer film is extracted with light petroleum in a Soxhlet apparatus to remove plasticisers and with toluene to remove resins. These are identified by well-known tests. Tritolyl phosphate is saponified only slowly with alcoholic KOH, and the plasticiser is therefore fused with KOH before testing for the cresol and phosphate formed.             D. R. Duncan

**3720. The analysis of organosilicon compounds.** Midland Silicones Ltd. Publ. A3–2, Feb. 1957, 7 pp.—A review is presented of current qual. and quant. methods for organosilicon compounds. Tests for detecting combined silicon and characteristic groupings are described, and the use of i.r. absorption is discussed. Procedures for the routine examination of finished products, namely silicone fluids, silicone resins and silicone rubbers, are suggested.                      G. S. Roberts

**3721. Absorptiometric determination of lead in rubber products and compounding materials.** K. E. Kress (Firestone Tire & Rubber Co., Akron, Ohio, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 803–807.—The sample (1 to 10 g) is decomposed by ignition or wet-ashing and the residue is dissolved in HCl $(1 + 1)$ (2 ml) and made up to 10 ml with the same acid. The absorption is measured at 250, 270 and 289 m$\mu$ *vs.* HCl $(1 + 1)$, and the concn. of $Pb^{2+}$ is derived from a given equation. Moderate concn. of $Bi^{3+}$, $Hg^{2+}$, $V^V$, $Cr^{3+}$, $Sn^{4+}$, $Sb^{3+}$, $Ca^{2+}$, $Mg^{2+}$, $Al^{3+}$, $Zn^{2+}$, $Co^{2+}$, $Ni^{2+}$ and $Mn^{2+}$ cause no interference, but $Cu^{2+}$ and $Fe^{3+}$ must be removed by preliminary treatment. Reproducibility data are presented.
                                   D. A. Pantony

See also Abstracts 3519, 3540, 3590, 3591, 3675, 3745, 3787, 3807.

**3722. Blood test [a detecting diagnostic composition in tablet form].** Miles Laboratories, Inc. Brit. Pat. 772,374; date appl. 23.12.54. U.S., date appl. 15.4.54.—The composition contains a compound which is colour-responsive to blood (o-tolidine, o-toluidine, benzidine or o-tolidine dihydrochloride), an oxidising agent of the peroxide type, an acetate, a water-sol. acid stronger than acetic acid, an effervescent mixture (*e.g.*, tartaric acid and $NaHCO_3$) and a water-insol. material (red, orange or yellow dye) which contrasts in colour with the colour formed by the colour-responsive compound in combination with blood. A typical composition comprises o-tolidine 1, $SrO_2$ 9, tartaric acid 30, Ca acetate 60, starch 5, talcum 5, effervescent mixture 10, and dye 0·1 pt. by wt. (*cf.* Brit. Pat. 708,990).                     J. M. Jacobs

**3723. Diagnostic agent.** Miles Laboratories, Inc. [Inventors: E. C. Adams and A. H. Free]. Brit. Pat. 779,303; date appl. 12.4.55.—A diagnostic composition for oral administration to determine gastric acidity comprises a cation-exchange resin complexed with 2:6-diamino-3-phenylazopyridine, the dye being liberated by the gastric acids and excreted in the urine.             N. E.

**3724. Diagnostic composition.** Miles Laboratories, Inc. Brit. Pat. 779,921; date appl. 6.4.55. U.S., date appl. 13.5.54.—A diagnostic composition for determining urobilinogen which comprises in dry form a $p$-dialkylaminobenzaldehyde and a solid organic acid, such as sulphosalicylic, maleic, tartaric, citric or oxalic, is claimed.       N. E.

**3725. A diagnostic composition for determining blood sugar concentration.** Miles Laboratories, Inc. Brit. Pat. 780,992; date appl. 16.11.54. U.S., date appl. 7.12.53.—A diagnostic composition is claimed comprising a mixture of a water-sol. cupric salt, $NaHCO_3$ and NaOH which may be formed into tablets. The heat of solution is sufficient to cause boiling and reduction of the cupric salt. The colour after the reduction is compared with a series of standards.                       N. E.

**3726. Estimation of cardiac output, independent of oxygen saturation of blood, in arterial block by means of a new dye absorbing in the near infra-red.** K. Kramer and G. Ziegenrücker (Physiol. Inst., Göttingen Univ., Germany). *Klin. Wochschr.*, 1957, **35** (9), 468–472.—The Evans blue method of Hamilton (*Amer. J. Physiol.*, 1932, **99**, 534) is modified by the use of a new dye termed "Rie 1743" that has a max. absorption at 800 m$\mu$.
                               H. F. W. Kirkpatrick

**3727. Determination of phosphate and sulphate in urine or blood by an ion-exchange method.** Teruo Asada (Physiol. Dept., Kyoto Prefectural Med. College, Kamigyo-Ku, Kyoto). *Japan Analyst*, 1957, **6** (2), 100–101.—Phosphate, pptd. as $UO_2HPO_4$, is converted into $H_3PO_4$ by treatment with Amberlite IR-120, in the presence of a negligibly small amount of $HNO_3$, the $UO_2^{2+}$ being adsorbed on the resin; $BaSO_4$ is similarly converted into an equiv. amount of $H_2SO_4$. The free acid obtained by this treatment is titrated with NaOH.
                                       K. Saito

**3728. A quick method for detection of thallium in urine in cases of poisoning.** F. Rappaport and F. Eichhorn (Beilinson Hosp., Petah Tikva, Israel). *Clin. Chim. Acta*, 1957, **2** (1), 16–20.—The Tl in 100 ml of urine is co-pptd. with CdS in alkaline soln. The washed ppt. is dissolved in HCl (1:1), the $H_2S$ is removed and Br soln. is added until the yellow colour is permanent. The excess of Br is removed by sulphosalicylic acid soln., then rhodamine B soln. in conc. HCl is added and a few ml of benzene. After shaking and centrifuging the mixture, a red fluorescent colour in the benzene layer indicates $>2$ μg of Tl. The reaction may be made semi-quant. by comparison with standards. Serum may be examined in the same way after boiling in acid soln. and filtering. No interference was observed when Sb, Fe or Hg were added to urine.
H. F. W. KIRKPATRICK

**3729. Colorimetric estimation of tolbutamide in serum.** H. Spingler (C. F. Boehringer and Soehne, Mannheim-Waldhof, Germany). *Klin. Wochschr.*, 1957, **35** (10), 533–535.—Shake 1 ml of serum with 5 ml of amyl acetate for 1 min., add 0·2 ml of N HCl and shake again for 3 min., then centrifuge for 2 min. To 4 ml of the supernatant amyl acetate soln. add 1 ml of 1-fluoro-2:4-dinitrobenzene reagent (0·1% v/v in amyl acetate), mix well and heat in a glycerol bath at 150° ± 1° for 5 min. Cool the product to room temp., first by allowing it to stand, and then by immersion in running water. Read the colour at 380 mμ against a blank prepared with 1 ml of water instead of serum. Prepare a standard soln. by dissolving tolbutamide in its equivalent of NaOH soln., dilute to obtain soln. containing 1·0 to 20·0 mg-%, and treat 1 ml of each soln. as for serum. Prepare a standard curve from the readings.          H. F. W. KIRKPATRICK

**3730. The photometric serum diastase estimation of Smith and Roe in clinical practice.** E. Kern, H. W. Lo, C. Flux, H. Schimmelpfennig and U. Billmann (Chir. Univ.-Klinik, Freiburg, Germany). *Klin. Wochschr.*, 1957, **35** (11), 572–576.—The original method (*cf. Brit. Abstr. C*, 1949, 340) is slightly modified and applied to various clinical conditions. High values were observed in about 20% of cases after operation on the upper abdomen; no correlation was found in the post-operative diastase and blood sugar, and no influence of anaesthesia or relaxation (curare) could be recognised. No correlation appears to exist between the serum diastase and malignancy.
H. F. W. KIRKPATRICK

**3731. Investigation of a test for vitamin-B₆ deficiency. Quantitative estimation of xanthurenic acid in urine after administration of DL-tryptophan.** H. Maske (Med. Univ.-Klinik, Munich, Germany). *Klin. Wochschr.*, 1957, **35** (11), 561–565.—After oral administration of 10 g of DL-tryptophan (**I**), normal individuals excrete an average of 10 to 20 mg of xanthurenic acid (**II**) during the next 14 hr. An excretion of 50 mg or more is evidence of vitamin-B₆ deficiency; if 5 g of **I** is given, the normal limit is 25 mg. **I** is administered at about 6 p.m. and the urine is collected until the next morning, including the first morning specimen. *Procedure*—Dilute 2 to 10 ml of filtered urine with $H_2O$ to 10 ml and mix with 10 ml of buffer soln. Pipette out 10 ml of the mixture to serve as the blank and to the other 10 ml add 0·1 ml of 1·7% aq. ferric ammonium sulphate soln. Read the green colour at 620 mμ after 5 min. against the blank and obtain the amount

of **II** from a standard curve covering the range 0 to 500 μg of **II** per ml. *Buffer soln.*—Dissolve 58 g of maleic acid and 60·6 g of tri(hydroxymethyl)-methylamine in 500 ml of $H_2O$, shake with 4 g of activated charcoal and filter. To 40 ml of filtrate add 48·4 ml of N NaOH and dilute to 100 ml with $H_2O$. The pH should be 7·8.
H. F. W. KIRKPATRICK

**3732. Serum cyanocobalamin level in chronic intestinal disorders.** M. J. Meynell, W. T. Cooke, E. V. Cox and R. Gaddie (Gen. Hosp., Birmingham, England). *Lancet*, 1957, **i** (18), 901–904.—The method described is essentially that of Thompson *et al.* (*cf. Brit. Abstr. C*, 1950, 415) modified by measuring the pH change instead of the turbidity as an estimate of the growth of the test organism (*Lactobacillus leichmannii*). The importance of washing the test organism carefully before inoculation is emphasised.          H. F. W. KIRKPATRICK

**3733. The evaluation of paper-electrophoresis strips with integration apparatus.** P. G. Scheurlen (Med. Univ.-Klinik, Tübingen, Germany). *Klin. Wochschr.*, 1957, **35** (9), 485–487.—The evaluation of the electrophoretic patterns of 15 different sera by integration ("Elphor-Integraph") showed a serious error when compared with planimetric measurements. A method of correcting the results is given.          H. F. W. KIRKPATRICK

**3734. The electrophoretic mobility of lipoprotein fractions of blood serum.** F. A. Pezold (Med. Klinik, Freien Univ., Berlin, Germany). *Klin. Wochschr.*, 1957, **35** (9), 475–476.—Electrophoresis of serum on agar gel gives three lipid fractions moving (*a*) with α-globulin, (*b*) with albumin and (*c*) faster than albumin. By this technique the β-globulin appears to be fat free. Fractions separated by ultracentrifugation show similar differences in their behaviour on filter-paper and agar. Absorption effects on filter-paper are suggested as a possible reason for the discrepancy in results.
H. F. W. KIRKPATRICK

**3735. Enzymatic estimation of urinary steroids.** B. Hurlock and P. Talalay (Ben May Lab. Cancer Res., Univ. Chicago, Ill., U.S.A.). *Proc. Soc. Exp. Biol. Med.*, 1956, **93**, 560–564.—The method depends on the selective oxidation or reduction of hydroxy- and keto-steroids, respectively, by highly purified hydroxysteroid dehydrogenases of bacterial origin. The method can be used for the determination of 3α-hydroxysteroids, 3β-(and 17β-)hydroxysteroids, 3-oxosteroids and 17-oxosteroids. N. E.

**3736. Metabolism and estimation of neutral steroids.** H. Rosenkilde and W. Schroeder (II Med. Univ.-Klinik, Hamburg-Eppendorf). *Z. Vitam.- Hormon.- u. Fermentforsch.*, 1956, **8** (4–5), 342–365.—A review is presented of the methods for the estimation and of the colour and fluorescence reactions of the ketosteroids and corticosteroids.
H. F. W. KIRKPATRICK

**3737. Micro-determination of acetylisable steroids in plasma.** D. L. Berliner (Univ. Utah, Coll. Med., Salt Lake City, U.S.A.). *Proc. Soc. Exp. Biol. Med.*, 1957, **94** (1), 126–128.—A combined paper-chromatographic - radioactive isotope technique gives high sensitivity and specificity not previously possible. Steroids with 3-, 20- or 21-hydroxyl groups are acetylated with radioactive [1-14C]-acetic anhydride. After chromatography, the

radioactivity of the steroid acetates is determined directly from the paper chromatogram. Plasma (5 to 10 ml) is extracted with $CHCl_3$, the solvent evaporated *in vacuo* and the fatty residue partitioned between hexane and 75% methanol. The methanol is separated, and evaporated, and the residue is extracted with $CHCl_3$. The extract is dried and acetylated with 700 $\mu g$ of radioactive acetic anhydride in pyridine overnight at room temp. This fraction contains the "free" steroids. Hydrolysis of the plasma residue with $\beta$-glucuronidase is carried out, and the hydrolysate is extracted with $CHCl_3$, evaporated and acetylated as before. The excess of acetic anhydride is removed by a stream of N. The dried residue is dissolved in $CHCl_3$ - methanol (1:1) and chromatographed by Zaffaroni's method. The amounts of the steroid acetates are determined from the radioactivity and area of the spots.                              N. E.

**3738. Steroid X-ray diffraction powder data.** J. Parsons, W. T. Beher and G. D. Baker (The Edsel B. Ford Inst. for Med. Res., Henry Ford Hosp., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 762–766.—X-ray diffraction powder data and powder-pattern photographs are presented for 32 steroids.                       E. G. CUMMINS

See also Abstract 3803.

## Drugs

**3739. Paper-chromatographic separation of the ergot alkaloids.** R. Schindler and A. Bürgin (Pharm. Inst., Univ., Berne). *Helv. Chim. Acta*, 1956, **39** (7), 2132–2136 (in German).—Two-dimensional chromatography is used. The papers (33 cm × 36 cm) are prepared by passing them through a 5% soln. of Dow Corning silicone 1107 in *cyclo*hexane, and drying, first at air temp. and then for 1 hr. at 110°. Excess of silicone is removed by elution with 6% formic acid in $CHCl_3$, in the same direction and under the same conditions as the first chromatogram, for about 8 hr. The air-dried paper is passed through formic acid - acetone (6:4), then suspended in air for 10 min. and chromatography is started [mobile phase, $CHCl_3$ - formic acid (9:1)]. The alkaloid soln. (methyl dichloroacetate is a suitable solvent) is placed at the starting point 2 to 7 min. after impregnation. A suitable amount is 10 to 20 $\mu g$; 50 $\mu g$ gives good separation; 1 $\mu g$ can be detected. The bands are located by u.v. fluorescence and cut out. The strips are placed over soda lime in a vacuum desiccator for 2 hr. Chromatography in the second dimension follows the method of Stoll and Rüegger (*Helv. Chim. Acta*, 1954, **37**, 1725). The papers are dipped from both ends up to the starting line in a 10% soln. of dimethyl phthalate in *iso*propyl ether, hung for 15 min. and chromatographed. The mobile phase [water - formamide (6:4)] must have a buffer capacity such that addition to 100 ml of 8 ml of 2 N formic acid changes the pH from 5 to 4. $R_F$ values are tabulated.                       G. BURGER

**3740. Use of oxycellulose in the assay of aqueous alkaloidal solutions for injection.** D. A. Elvidge, K. A. Proctor and C. B. Baines (Boots Pure Drug Co. Ltd., Standards Dept., Nottingham, England). *Analyst*, 1957, **82**, 367–372.—Oxycellulose powder (16 to 22% of carboxyl) can be used as a carboxylic cation-exchange medium for the separation of the active ingredient and the bacteriostatic agent in aq. soln. for injection. The oxidised cellulose is formed into a column with $N$ $H_2SO_4$, and the column is washed with water until free from acid. The injection soln. is applied to the column, which is then eluted with water. The bacteriostatic agent in the suitably diluted eluate is determined spectrophotometrically at 269 m$\mu$ for phenol or 279 m$\mu$ for chlorocresol. The active ingredient is then eluted with $N$ $H_2SO_4$ and determined spectrophotometrically. The extinctions of several alkaloidal and other active ingredients are listed. The recovery of the active agents from 17 different injection soln. ranged from 96·4% to 102% when phenol was present, and from 97·4% to 106% when chlorocresol was present as the bacteriostatic agent.                              A. O. JONES

**3741. Estimation of the component cardiac glycosides in digitalis plant samples. III. Separation and estimation of the genins and anhydrogenins.** H. Silberman and R. H. Thorp (Dept. of Pharmacology, Univ. Sydney, Australia). *J. Pharm. Pharmacol.*, 1957, **9** (6), 401–405.—Complete separation of 20 to 30-$\mu g$ quantities of the anhydrogenins of digitalis is readily achieved by chromatography on Whatman No. 3 MM paper with $CHCl_3$ - benzene - $H_2O$ (2:8:5) as mobile phase. Separation of gitoxigenin and digoxigenin is less satisfactory, although both of these are readily separated from digitoxigenin; $CHCl_3$ - benzene - $H_2O$ (8:2:5) is preferred as mobile phase for separation of the genins.                              A. R. ROGERS

**3742. The assay of oxytocin in the presence of vasopressin on the dioestrus uterus of the rat.** M. F. Lockett and G. E. Owen (Dept. Physiol. Pharmacol., Chelsea Poly., London). *J. Pharm. Pharmacol.*, 1957, **9** (6), 406–414.—The effect of variation in $Mg^{2+}$ concn. on the dose - response curves given by oxytocin (**I**) and vasopressin (**II**) on the dioestrus uterus of the rat was similar whether these hormones were examined alone or in a mixture. Max. sensitivity was found to **I** at 0·005% of $MgCl_2$ and to **II** at 0·05% of $MgCl_2$. Changes in sensitivity were caused by change in position but not in slope of dose - effect curves. Increase in concn. of $CaCl_2$ from 0·003 to 0·012% caused slight increase in sensitivity towards **I**, but no change towards **II**. Conditions under which the rat's dioestrus uterus may be used to give reliable estimates of the **I** present in 1:1 mixtures with **II** have been defined. Concn. of 0·0001% of $MgCl_2$ and 0·012% of $CaCl_2$ are recommended.                              A. R. ROGERS

**3743. Estimation of chloramphenicol cinnamate.** F. A. Robinson, M. E. Wright and J. R. Whittingham (Allen and Hanburys Ltd., Ware, Herts.). *J. Pharm. Pharmacol.*, 1957, **9** (5), 320–325.—Chloramphenicol cinnamate (**I**) and its aq. suspension can be assayed by enzymic hydrolysis to chloramphenicol, followed by microbiological determination with *Sarcina lutea*. *Procedure*—Dissolve **I** (10 mg) in ethanol (4 ml) and add the soln. to an extract prepared by grinding fresh rat liver (1 g) with sterile $H_2O$ (94 ml). Adjust the pH to 7·2 by addition of 0·1 $N$ NaOH and dilute to 100 ml with sterile $H_2O$. Add $CHCl_3$ (1 ml), immerse in a water bath at 37° and stir continuously for 24 hr. Assay by the cup-plate method with *S. lutea* as test-organism and pure chloramphenicol as standard. If the sample is a 4·5% (w/v) aq. suspension of **I**, shake the sample (5 g) with ethanol (50 ml), allow to stand and use a 2-ml aliquot of the clear supernatant liquid for the assay.                              A. R. ROGERS

**3744. The poison-producing mould isolated from dry malt. VI. Micro-detection and determination of patulin.** Takeo Yamamoto (Kobe Munic. Hyg. Lab., Higashiyama-cho, Hyogo-ku, Kobe). *J. Pharm. Soc. Japan*, 1956, **76** (12), 1375–1377.— The paper-chromatographic micro-detection of patulin (**I**) ($>2$ $\mu g$) (developer, 1 or 5% aq. phenol soln.) was studied by the use of its colour reaction with phenylhydrazine (orange - yellow) (*Ibid.*, 1954, **74**, 450). This reaction is also made quant. by preparing standards. **I** in cultures is adsorbed on activated charcoal, extracted with 80% acetone and evaporated, and the residue is chromatographed.      K. SAITO

**3745. Determination of benzoic acid and salicylic acid mixtures by differential non-aqueous titration.** M. I. Blake (School of Pharmacy, North Dakota Agric. Coll., Fargo, N.D., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (5), 287–290.— The differential titration of benzoic acid and salicylic acid has been examined with a mixture of the two acids free of added ingredients, and with mixtures of the acids in the form of ointment. The acids were in soln. in dimethylformamide and the titrant was Na methoxide.      W. J. WRIGHT

**3746. Colorimetric determination of diazotisable amines (procaine, benzocaine, sulphonamides) by coupling with thymol.** E. Zöllner and G. Vastagh (Chem. Abteilung, Staatl. Inst. für Hyg., Budapest). *Pharm. Zentralh.*, 1957, **96** (5), 199–206.—*Procedure* —Mix the sample soln. (5 or 10 ml containing 20 to 200 $\mu g$ of amine) with 10% HCl (2·5 ml). Cool in ice and add freshly prepared 0·5% NaNO₂ soln. (5 ml). After 5 min., add urea (1 g) and shake during 15 min. at room temp. Add freshly prepared 0·5% thymol in 10% NaOH soln. (1 ml) and 10% NaOH soln. (5 ml). After 10 min., dilute with H₂O to 100 ml and measure the extinction against a reagent blank in 2-cm cuvettes with filter S47. Modifications allow for the assay of tablets, injections and other pharmaceutical preparations.      A. R. ROGERS

**3747. Determination of 2-[di-(N-acetylsulphanilyl)]-aminothiazole in the commercial product (Prontyl).** Z. Bellen and Z. Mroczkowska (Anal. Dept., Inst. of General Chemistry, Warsaw). *Chem. Anal., Warsaw*, 1956, **1** (4), 320–330.—The method is based on ammonolysis of 2-[di-(N-acetylsulphanilyl)]-aminothiazole (**I**) to a mixture of acetylsulphathiazole and N⁴-acetylsulphanilamide; the former is then titrated potentiometrically with alkali. *Procedure*—To the sample (2 g) add ethanol (20 ml) and conc. aq. NH₃ (20 ml). Evaporate to dryness by heating on a boiling-water bath. To the residue add ethanol (10 ml) and conc. aq. NH₃ (10 ml) and again evaporate. Dissolve the residue in acetone - water (4 : 1) (150 ml), titrate with 0·2 N aq. NaOH and determine the end-point by means of an antimony electrode and a calomel electrode in conjunction with a potentiometer. Carry out a blank determination, on another 2-g portion of the sample, by direct titration with 0·2 N NaOH in the acetone - water mixture. Subtract the latter titration value from that obtained in the first titration and calculate the content of **I** by assuming 494·56 to be its mol. weight. The method is unaffected by impurities usually present in the technical material. The accuracy of results obtained with pure and commercial samples was within ±2%.      K. F. SPOREK

**3748. Estimation of formaldehyde in poliomyelitis vaccines.** E. M. Taylor and P. J. Moloney (Connaught Med. Res. Lab., Univ. Toronto). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (5), 299–301.— Nash's method (*Biochem. J.*, 1953, **55**, 416) has been investigated, and its validity in determining formaldehyde in vaccines containing no bisulphite has been established. A modification of the method for use with vaccines to which bisulphite has been added gives results of the same order as those obtained by vapour-equilibrium technique.      W. J. WRIGHT

See also Abstracts 3692, 3729, 3765.

## Food

**3749. Lead error in polarisation of raw cane sugars.** Anon. *Int. Sugar J.*, 1956, **58**, 337; 1957, **59**, 14.—Full tables for wet and dry lead error corrections in the polarisation of raw cane sugars are reproduced. The wet and dry lead compositions were defined at the 11th Session of I.C.U.M.S.A. The wet lead consists of basic Pb acetate soln. prepared by dissolving Pb subacetate (A.C.S. specification) and adjusting the sp. gr. to 1·25. The dry reagent (A.C.S. specification) is, if necessary, ground to the fineness of the Australian specification: 100% passing through a sieve with aperture 0·0164 in. square (35-mesh Tyler scale) and 70% through a sieve with aperture 0·0049 in. square (115-mesh Tyler scale). For the A.C.S. specification, see *Ind. Eng. Chem.*, 1944, **16**, 282. Owing to the finding of different effects by the Colonial Sugar Refining Co. (concerning the basicity of the Pb acetate) these tables will, however, not yet be put into force by the British National Committee of I.C.U.M.S.A., as previously intended.      SUGAR IND. ABSTR.

**3750. Comparison of some methods of invert sugar determination in the presence of sucrose.** L. Rozental. *Roczn. Zakl. Hig., Warsaw*, 1956, **7**, 521–530.—Comparative tests of the Lane - Eynon, Luff - Schoorl, Bruhns, Bertrand, and Fellenberg and Demont methods have shown that the Lane - Eynon method is best; the Luff - Schoorl method is sufficiently accurate when the sucrose content is $<99\%$; the Bruhns method is suitable only for approximate determinations; the last two methods were unsatisfactory.      SUGAR IND. ABSTR.

**3751. Nephelometric method for determination of invert sugar in white sugar and refined sugars.** T. Kozakiewicz. *Gaz. Cukr.*, 1956, **58**, 299–301.— The method is based on the reduction of HgI₂ (in KI soln.) to bright yellow HgI, which has a very low solubility and which can be measured nephelometrically. Qualitatively, 0·00025% of invert sugar can be detected; quantitatively, 0·001% can be determined. For qual. testing, 50 ml of water and 10 ml of N NaOH are heated to just over 50°, 20 g of refined sugar is added and the mixture shaken and cooled to 45°; 10 ml of a 1% soln. of HgI₂ in KI is added, mixed quickly and the mixture is set aside in the dark. In the presence of invert sugar a cloudy yellow suspension appears; if too much is present the yellow turns to green then grey. If the invert sugar content is very low, observation should be continued for 15 min. For quant. determination the sample should contain $> 8$ mg of invert sugar in 20 g. The procedure is the same up to the HgI₂ addition; the flask is then kept in a thermostat

at 25° for 20 min. The measurement can then be made in a nephelometer or by use of a Helmer cylinder; the results are compared with a graph prepared from measurements of a series of known soln. SUGAR IND. ABSTR.

**3752. Determination of saponins in refined beet sugars.** H. G. Walker, jun. *J. Amer. Soc. Sugar Beet Technol.*, 1956, **9**, 233–237.—The saponins are pptd. from sugar soln. by acidification and filtration, and are then determined colorimetrically in glacial acetic acid soln. with $SbCl_5$ in $CHCl_3$, which gives a red complex with an absorption max. at 535 m$\mu$. Dissolve 125 g of sugar in 250 ml of water, and add 2·5 ml of conc. HCl. Filter in a Buchner funnel, using 1·25 g of pre-coated filter-aid and 3·75 g of filter-aid in the soln. Wash the ppt. with acidified water, and dry for 20 min. at 105°. Dissolve in 8 ml of hot glacial acetic acid, and make up to 10 ml. To a 1-ml aliquot in a colorimeter tube add 7 ml of 10% $SbCl_5$ reagent. Mix, allow to stand for 10 min. (max. colour formation), and measure the absorbance at 535 m$\mu$. The saponin content is then calculated from a graph of values for a standard saponin reagent, and a blank determination. The standard saponin is prepared from factory juices or lime muds (*cf. Sugar Ind. Abstr.*, 1953, 31, 263), making a 0·04% soln. in glacial acetic acid, which keeps well. The $SbCl_5$ reagent should be made chlorine-free by vacuum distillation, or addition of 10 mole-% of $SbCl_3$, or by blowing dry air through the soln. The accuracy of the method should be sufficient for control work. SUGAR IND. ABSTR.

**3753. Conductimetric determination of ash in clear syrups.** Z. Frimlová and M. Friml. *Listy Cukr.*, 1957, **73**, 105–106.—Methods for the conductimetric determination of ash in diluted syrups are described. Tables and graphs are given of the results of measurements in raw sugar and white sugar soln. and in mixtures. As little as 0·03% of ash can be recorded. SUGAR IND. ABSTR.

**3754. Chromatographic method of determination of iron in molasses.** S. Zagrodzki and Z. Olszenko-Piontkowa. *Gaz. Cukr.*, 1956, **58**, 281–282.—The sample (5 g) in 20 ml of water is passed at 2 ml per min. through a column of 20 g of cation-exchange resin, which is then washed until free from colour. The column is eluted with 95 ml of 2 N HCl and the Fe in the eluate is oxidised and determined colorimetrically with thiocyanate. The method is accurate to ±0·2 mg when the Fe present is <1·0 mg. SUGAR IND. ABSTR.

**3755. Determination of acetyl in pectin and acetylated carbohydrate polymers. Hydroxamic acid reaction.** E. A. McComb and R. M. McCready (U.S. Dept. Agric., Albany, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 819–821.—The method is based on the formation of acetohydroxamic acid (**I**) by the interaction of the secondary acetyl groups of pectin with alkaline hydroxylamine. **I** forms with $Fe^{3+}$ a soluble red complex, which is determined colorimetrically. Pectin hydroxamic acid, produced from the reaction of pectin with hydroxylamine, forms an insol. red complex with $Fe^{3+}$, which is removed by filtration. The concn. range of the method is ≃80 to 450 $\mu$g of acetyl per ml, and the accuracy is within ≃±2%. These reactions also apply to the quant. determination of acetyl in acetylated carbohydrate polymers. G. P. COOK

**3756. Detection of orange peel in orange drink (comminuted).** R. Born (J. Lyons & Co., Ltd., London). *Chem. & Ind.*, 1957, (23), 734–735.—Drink made with whole oranges may be distinguished from that made with juice only by chromatography of a benzene extract of the drink on an alumina column, followed by spectrophotometry of a defined fraction. The column is washed with 0·5% *iso*propyl alcohol in light petroleum until a sharp yellow-orange band, and an intense violet band (visible in u.v. only), have been washed out. The column is then eluted with a soln. of acetone in light petroleum, and the intense violet band thus developed is collected. The eluate is evaporated to dryness and taken up in $CHCl_3$, and the extinction coeff. at 325 m$\mu$ is measured. For squashes made from juice only, the coeff. was $\not> 0·25$, and usually $< 0·2$. Drinks from whole fruit gave readings of 0·3 to 0·9. W. J. WRIGHT

**3757. Chromatographic determination of trace amounts of sucrose in beer.** D. A. M. Mackay and R. L. Evans (Evans Research and Development Corp., 250 East 43rd St., New York, U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (4), 298–300.—In a quart sample of bottled beer, after removal of the proteins, the ethanol concn. were brought successively to 67, 80 and 85% and the deposited syrups were examined. The sol. material in the 85% alcoholic syrup was evaporated to dryness, extracted with pyridine to separate the salts, and the pyridine removed under vacuum. The residue was dissolved in 80% ethanol, then filtered from gummy material, and examined by chromatography alongside known amounts of sucrose. The spots were detected with 1 : 3-dihydroxynaphthalene - trichloroacetic acid. In tests of validity with known amounts of added sucrose, at concn. >10 p.p.m., the sucrose was easily observable; at 7·5 p.p.m. it was just observable. At lower concn., materials present in the filter-paper affected the result. S.C.I. ABSTR.

**3758. Quantitative paper-chromatographic determination of malic acid [in wine].** G. Lehongre, H. Tanner and H. Rentschler (Eidg. Versuchsanstalt f. Obst.-, Wein- u. Gartenbau, Wädenswil, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (1), 40–46.—The malic acid is separated by passage through a cation-exchange resin (Dowex 50 or Amberlite IR-120), and the eluate is chromatographed on paper. The spots are sprayed with an acridine soln., revealed under u.v. light, and cut out and weighed. Spots obtained with known quantities of malic acid are used for comparison. The method has an accuracy within ±5% and is suitable for wines with a sugar content up to 10 g per litre. Other organic and inorganic acids may be estimated simultaneously. W. J. WRIGHT

**3759. Rapid spectrophotometric technique for evaluation of vanilla extracts.** R. Pomerantz, S. A. Goldblith and B. E. Proctor (Dept. Food Technol., M.I.T., Cambridge, Mass., U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (4), 292–293.—Transmittance data were obtained for various dilutions, in water or 37% ethanol, of vanilla samples (4 pure, 7 artificial) at the critical wavelength of 700 m$\mu$. When water was used to dilute the soln., only the artificial vanillas produced a straight-line relation (percentage of transmission at 700 m$\mu$ plotted against dilution) identical with that of the alcohol dilution in slope and intensity of transmittance. Curves for pure vanilla are distorted at water

dilutions of 0 to 40%. The rapid scanning spectrophotometer was used to differentiate between 60% soln. of samples of pure and artificial vanillas.

S.C.I. ABSTR.

**3760. Colorimetric determination of ethylene oxide [in spices] by conversion to formaldehyde.** F. E. Critchfield and J. B. Johnson (Carbide and Carbon Chemicals Co., S. Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 797–800.—The method is based on the hydrolysis of ethylene oxide to ethanediol, which is treated with $NaIO_4$ to yield formaldehyde. The excess of periodate is reduced by $Na_2SO_3$ and the formaldehyde is determined colorimetrically with Na chromotropate in conc. $H_2SO_4$. The ethylene oxide can be removed from interfering substances by evolution from boiling water. The method was applied to the analysis of spices fumigated with the oxide; essentially complete recovery was obtained over the range of 6 to 136 p.p.m. of added ethylene oxide, and the limit of detection was $\simeq 1$ p.p.m.    G. P. COOK

**3761. Determination of iodine value.** A. Seher and W. Arends (Dtsch. Inst. f. Fettforschung, Univ. Münster, Westfalen). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (1), 1–4.—Comparative determinations of the iodine value of various vegetable oils, fatty acids and fish-liver oils were carried out by Stähli's modification of the Wijs method (*cf. Anal. Abstr.*, 1955, **2**, 3212) and by Kaufmann's method. In every case, values increased with increasing times of reaction, from 2 to 24 hr., but least with the Kaufmann procedure. The values after 2 hr. by this method were in agreement also with those obtained by hydrogenation. The superiority of the Kaufmann method is thus established.

W. J. WRIGHT

**3762. Determination of iodine value by the Wijs method.** H. Sulser (Lab. d. Eidg. Gesundheitsamtes, Bern). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (1), 5–11.—Stähli's modification of the Wijs method comprised the use of excess of iodine (7 g per litre) whereby the increase in the iodine value, due to an increase in the reaction time from 2 to 24 hr., is avoided. In an investigation of the test, the soln. was prepared in accordance with Stähli's prescription but, of six oils tested, only three showed a constant iodine value when the reaction time was increased from 2 to 24 hr. The actual excess of iodine was found to be 5·6 to 5·7 g per litre, probably owing to variations in the composition of commercial iodine trichloride. When the excess was made up to 7 g per litre by adding iodine, constant iodine values were obtained after a 24-hr. reaction time, even for oils with high iodine values such as linseed oil.    W. J. WRIGHT

**3763. Estimation of saturated acids in mixed fatty acids of natural fats.** R. G. W. Spickett, E. F. J. Thorpe and J. B. Ward (Colonial Prod. Lab., Imperial Inst., London). *Chem. & Ind.*, 1957, (23), 734.—The methyl esters of the mixed fatty acids are oxidised with $KMnO_4$ in acetone soln. and the oxidation products are dissolved in light petroleum (boiling range 40° to 60°). The soln. is percolated through a column of alumina, and the saturated methyl esters, collected from the eluate after removal of the solvent, are weighed. The amounts of saturated acids obtained from various fats are tabulated and compared with those obtained by other methods. They are generally

higher than those shown by Bertram's method or calculated from iodine and thiocyanogen values.

W. J. WRIGHT

**3764. Quantitative determination of the antioxidants propyl, octyl and dodecyl gallate in oils and fats.** H. J. Vos, H. Wessels and C. W. T. Six (Central Inst. for Nutrition Res., T.N.O., Utrecht, The Netherlands). *Analyst*, 1957, **82**, 362–367.—The method described is a modification of the colorimetric method of Mahon and Chapman (*Brit. Abstr. C*, 1952, 25) for propyl gallate, which is based on the colour reaction with ferrous tartrate specific for polyphenolic compounds, and used by Mattil and Filer (*Brit. Abstr. B II*, 1944, 365) for the determination of gallic acid in oils and fats. Propyl gallate is extracted from a soln. of the sample in light petroleum with water. An aliquot of the extract is heated with ferrous tartrate soln., the mixture is adjusted to a definite vol. with a Na acetate buffer soln., and the extinction is measured against water at 530 mμ. After the propyl gallate has been extracted from the light petroleum soln., octyl and dodecyl gallate are extracted with abs. methanol. The methanol extract is treated with ferrous tartrate and Na acetate, and the coloured complex is extracted with *iso*amyl alcohol - light petroleum, and is measured absorptiometrically at 550 mμ. A method of distinguishing the two gallates is given.    A. O. JONES

**3765. Microbiological assay of vitamin $B_{12}$ in presence of methionine.** S. S. Ghosh and S. K. Dutta (Bengal Immunity Res. Inst., Calcutta). *Indian J. Pharm.*, 1957, **19** (4), 96.—In estimating vitamin $B_{12}$ in the presence of methionine or its derivatives, De-Acidite FF (OH form) was used to adsorb the methionine, the cyanocobalamin in the eluate being then assayed microbiologically with *Escherichia coli* mutant 301. The adsorbed methionine could be eluted with dilute NaOH soln. and estimated by Horn's method. The recovery of the methionine was 98 to 99% and of vitamin $B_{12}$ was 99%. A spectrophotometric estimation gave similar results.    W. J. WRIGHT

See also Abstract 3760.

## Sanitation

**3766. Specific method of analysis for copper in air.** N. P. Anashkina. *Symposium:—Voprosy̌ Gigieny̌ Truda Professional. Patolog. i Toksikolog. v Prom. Sverd. Obl. Sverdlovsk*, 1955, 249–254; *Ref. Zhur., Khim.*, 1956, Abstr. No. 78,425.—For the determination of copper vapour in the presence of vapours of Pb, Zn, Sn and other metals, use is made of the reaction with 4-phenyl-1:2-dithiole-3-thione (*cf.* Voronkov and Tsiper, *Zhur. Anal. Khim.*, 1951, **6**, 331). The air is drawn at a rate of 10 litres per min. through glass wool moistened with 3 to 5 ml of 3% $HNO_3$, a total of 500 to 1000 litres being passed. The absorbed Cu is washed from the glass wool with 3% $HNO_3$, and water if necessary, the soln. is evaporated down, and a drop of the liquor transferred to a dry reaction paper, saturated with an ethanolic soln. of 4-phenyl-1:2-dithiole-3-thione. In the presence of Cu, a pink spot is obtained, the intensity of which is compared with standard spots prepared from soln. of concn. 0·001, 0·0025, 0·005, 0·0075 and 0·01 mg of Cu per ml. The determination is possible also at a concn. of 1 μg

of Cu per ml. No colour is obtained with up to 0·1 mg per ml of Pb, Zn or Mn. With $HNO_3$ concn. of 10%, the spot becomes greyish. C. D. KOPKIN

**3767. Rapid determination of formaldehyde in air.** V. P. Fedotov. *Gigiena i Sanit.*, 1956, (9), 87–89; *Ref. Zhur., Khim.*, 1957, Abstr. No. 15,916.—The method is based on the absorption of the formaldehyde in a layer of silica gel (**I**) steeped in phenylhydrazine hydrochloride and the formation of a red colour on adding an oxidising agent. To prepare the indicator tubes, steep **I** (of grain diam. 0·25 to 0·6 mm) in a freshly prepared 0·3% alcoholic soln. of phenylhydrazine hydrochloride, using 0·5 ml of reagent for 0·5 g of **I** and mixing till quite dry. Insert the material obtained into glass tubes (diam. 5 mm, length 6 cm) to give a layer 2 cm long, packed between cotton plugs. The tubes may be used for 8 to 10 hr. The oxidising soln. is prepared by dissolving 0·04 g of $K_3Fe(CN)_6$ in 10 ml of HCl (sp. gr. 1·12), and is stable for three to four days. The air is sucked through the tube at a rate of 1 litre in 2·5 min. The colour formed is compared with coloured standards. No interference is caused by vapours of acetaldehyde, acraldehyde, furfuraldehyde, acetone, phenol, $H_2S$, or $SO_2$ in concn. $\gg 0·1$ mg per litre. C. D. KOPKIN

**3768. Application of photometry to water analysis.** J. W. Polsky. *Bull. Amer. Ceram. Soc.*, 1956, **35** (11), 433–436.—A photo-electric photometer was used to determine up to 40 p.p.m. of $SO_4{}^{2-}$ with an accuracy within $\pm 0·3$ p.p.m. A turbidimeter cell (20 mm × 20 mm) and a 415-m$\mu$ filter were employed. *Procedure*—A reference soln. contains 50 ml of sample, 5 ml of an aq. soln. of 240 g of NaCl and 20 ml of conc. HCl per litre, and 10 ml of 50% (v/v) glycerol soln. The test soln. contains 50 ml of sample, 5 ml of NaCl - acid soln. and $\simeq 0·2$ g of $BaCl_2$. The soln. is stirred until the $BaCl_2$ is dissolved, set aside for 5 min., then stirred, and the extinction is measured within 1 min. The photometer is calibrated with known amounts of $SO_4{}^{2-}$. The zero and a standard in the middle of the range should be checked before each measurement.

J. A. SUGDEN

**3769. Flame photometry in the analysis of natural waters. II. Determination of potassium.** P. Valori and F. Savoini (Ist. Igiene, Univ. Roma). *Ric. Sci.*, 1957, **27** (4), 1204–1215.—This note reports an extension of previous work (*cf. Ibid.*, 1957, **27**, 791), and gives the method of adapting the test for the determination of Na in natural waters to the determination of K. The only difference lies in the solution used to reduce and stabilise interference effects, which is prepared from chlorides of the interfering elements, specially checked for purity as regards potassium content. The interfering substances considered are Na, Li, Ca, Mg and Sr. It is claimed that the method is satisfactory for the determination of K in concn. as low as 0·1 to 0·05 p.p.m. J. F. P. H. GREENE

**3770. Quantitative determination of organic nitrogen in water, sewage and industrial wastes.** G. B. Morgan, J. B. Lackey and F. W. Gilcreas (Univ. of Fla., Gainesville, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 833–835.—A modified Kjeldahl method, in which $H_2SO_4$ - $HgSO_4$ - $K_2SO_4$ is used as the digestion mixture, is described. An average recovery of N of 98·5% with a standard deviation of $\pm 0·235\%$ was obtained from amino-acid standards. Results were in good agreement with those of another

Kjeldahl procedure when the method was applied to some primary effluent samples. Carbohydrates and lipids do not appreciably affect the analysis and no appreciable loss of organic N results from the presence of $NO_3{}^-$ and $NO_2{}^-$. G. P. COOK

**3771. Colorimetric determination of cadmium, chromium, copper, iron, lead, manganese, nickel and zinc in sewage and industrial wastes.** A. A. Christie, J. R. W. Kerr, G. Knowles and G. F. Lowden (Gov. Lab., Dudley House, Endell St., London). *Analyst*, 1957, **82**, 336–342.—Preliminary treatment consists in wet oxidation of the conc. sample with $HNO_3$ and $HClO_4$, followed by ignition at 300° and solution of the residue in HCl. Insol. matter is $SiO_2$ or $PbSO_4$. Chromium is determined spectrophotometrically (540 m$\mu$) with diphenylcarbazide after oxidation to $Cr_2O_7{}^{2-}$. Copper is determined spectrophotometrically (650 m$\mu$) by means of the colour given with dithio-oxamide in the presence of malonic acid and ammonium acetate. Iron is determined spectrophotometrically ($\simeq 535$ m$\mu$) by means of the colour with thioglycollic acid. Manganese is oxidised to $MnO_4{}^-$ and determined spectrophotometrically (525 m$\mu$). The colours in these methods may also be measured visually. Zinc is determined by a slight modification of the mono-colour dithizone procedure of Sandell ("Colorimetric Determination of Traces of Metals," 2nd Ed., Interscience Publ. Inc., New York, 1950). Details of these methods are given. Methods published for Cd (Saltzmann, *Brit. Abstr. C*, 1953, 291), Pb and Ni (Sandell, *loc. cit.*) are recommended without modification.

A. O. JONES

See also Abstracts 3628, 3787.

### Agriculture and Plant Biochemistry

**3772. Systematic determination of inorganic elements in plant tissues. I. Decomposition of the sample.** Noboru Yamagata, Satoru Muto, Toshiko Yamagata and Sadakata Watanabe (Kiriu Coll. Technol., Gunma Prefecture). *Japan Analyst*, 1957, **6** (2), 75–77.—Total analysis of 12 elements in various plant tissues was made in order to find the most suitable method of decomposition of the sample. For the determination of K, Ca, Mg, Na and P, decomposition with $HNO_3$ (2 ml for 0·2 g of dried sample) and $HClO_4$ is the best; Si, S and Cl are determined after fusion with $Na_2CO_3$ and $Na_2O_2$.

**II. Separation and determination of sodium and potassium by ion exchange.** Sadakata Watanabe and Noboru Yamagata. *Ibid.*, 1957, **6** (2), 97–99.—Sodium and K (both $< 6$ m$M$) are successively eluted from Dowex 50 (H) with $N$ HCl (the first 90 ml for Na, and the following 180 ml for K). The potassium fraction is free from Ca, provided that the rate of flow is 0·8 to 1·0 ml per min.; Mg is found in this fraction, but is converted into MgO on evaporating the eluate. The sample, after being decomposed by $HNO_3$ and $HClO_4$, is dissolved in HCl and passed through a column (diameter 1 cm, height 15 cm) of Dowex 50. The eluates are evaporated to dryness, heated in an air bath (150°) for 30 min., dissolved in water and titrated with 0·02 $N$ $AgNO_3$. The reproducibility compares favourably with that of the chloroplatinate method. K. SAITO

**3773. Chemical analysis of trace elements in biological materials. XL. Analysis of plant ash by a polarographic method. (4). Use of co-precipitation for the concentration of some ions.** Katsumi

Yamamoto (Chem. Dept., Fac. of Sci., Tohoku Univ., Sendai). *Japan Analyst*, 1957, **6** (2), 94–97.—Copper (<1 mg per g of the ash), Zn (<1 mg) and Mn (<1 mg) are co-pptd. with Fe(OH)$_3$ on treatment of their HCl soln. with dil. aq. NH$_3$ soln. to make the pH $\simeq 7\cdot 4$. For plants containing small amounts of Fe, addition of Fe ($\simeq 10$ mg) is recommended. The pptd. hydroxides are dissolved in HCl and concentrated by cation-exchange resin; the Fe is extracted as FeCl$_3$ with ether and the soln. is polarographed in tartaric acid - NH$_4$Cl - aq. NH$_3$.
K. SAITO

**3774. Use of sodium peroxide fusion for the determination of fluorides in vegetation.** R. Mavrodineanu and J. Gwirtsman (Boyce Thompson Inst. for Plant Res., Inc., Yonkers 3, N.Y., U.S.A.). *Contr. Boyce Thompson Inst.*, 1956, **18**, 419–420.—The dried, finely ground sample (0·5 g) is placed in the fusion cup of a 22-ml Parr bomb and 0·5 g of sucrose and 0·5 g of finely ground accelerator (KNO$_3$ or KClO$_4$) are added with about 15 g of Na$_2$O$_2$. Ignition is carried out in the usual manner, followed by extraction of the fused mass and determination of F by distillation and titration. The fusion time is 30 min. as compared with the 6 hr. required for ashing with CaO in the standard procedure.
E. G. BRICKELL

**3775. Sulphur in soils. II. Determination of the total sulphur content of soil.** R. C. Little (Nat. Agric. Advisory Service, Wye, Kent, England). *J. Sci. Food Agric.*, 1957, **8** (5), 271–279.—Methods for the determination of total S in soil are reviewed, and a new procedure is described in which all the S is reduced to sulphide by heating the sample (0·5 to 1 g) with reduced iron (5 g) in an electric furnace at 700° to 900°. The sulphide is treated with dil. HCl and the H$_2$S is determined iodimetrically after absorption in 0·02 N potassium hypochlorite. The method, which is specific for S (As and P do not interfere), is applicable to all types of soil and also to relatively pure organic and inorganic compounds containing S. The mean error is $\pm 2\cdot 5\%$. With four sets of apparatus, eight samples can be analysed per day.
W. J. BAKER

**3776. Application of the Karl Fischer method to grain moisture determination.** J. R. Hart and M. N. Neustadt (Grain Div., Agric. Marketing Service, U.S. Dept. of Agric., Washington, D.C., U.S.A.). *Cereal Chem.*, 1957, **34** (1), 26–37.—Results of the titrimetric method described agree well with those obtained by official oven-drying methods for wheat, oats, barley, rice and rye, but are slightly low for soya-beans and flax-seed, and slightly high for pea beans and maize. *Procedure*—The sample (10 g) is ground for 3 min. in a suitable mill fitted with a cover through which a water-cooled condenser is attached to prevent loss of solvent during subsequent grinding; a drying tube is attached to the open end of the condenser. After adding 200 ml of methanol through the condenser tube, the grinding is continued for a further 5 min. The slurry is cooled by immersing the mill container in cold water, and the liquid is decanted and the suspended matter allowed to settle (5 to 10 min.). A 20-ml aliquot is diluted with 40 ml of methanol and titrated with Karl Fischer reagent by the dead-stop back-titration method.
S. C. JOLLY

**3777. Determination of carotene in silages and forages.** H. G. Wiseman, H. M. Irvin and L. A. Moore (Dairy Husbandry Res. Branch, U.S. Dept. of Agric., Beltsville, Md., U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (2), 134–137.—A rapid chromatographic method is described. *Procedure*—The chopped sample (50 g for maize silage, 20 g for fresh green forage and grass or legume silage, and 10 g for lucerne meal or ground hay; the last should be mixed with 30 to 35 ml of water at 60° to 70°) is mixed in a Waring Blendor with 275 ml of foaming mixture (ethanol - Skellysolve B) (3:1, by vol.) and 25 ml of methanolic KOH (1 lb of KOH in 1400 ml of methanol), gently at first until max. foaming conditions are established. The foaming conditions and splash-free blending are established by the addition of either dehydrated ethanol (when foaming is absent) or water, and then at full speed for 10 min. The homogenate is filtered with suction through a thin layer of Celite 535 directly into a separating funnel, and the blender and residue are washed first with dehydrated ethanol and then with 170 ml of Skellysolve B. The combined filtrate and washings are shaken thoroughly with 350 ml of 4% w/v aq. NaCl soln. and allowed to separate, and the upper layer is added to 6 ml of acetone. The mixture is diluted to 200 ml with Skellysolve F, and 10-ml aliquots are chromatographed on a column (15 g of a mixture of equal parts of MgO and Celite 535 slurried in Skellysolve B and transferred to a 25-mm-diam. tube), $\simeq 75$ ml of eluting solvent (acetone - Skellysolve B) (9:91, by vol.) being forced through the column under pressure of N. The yellow fraction of the eluate is diluted to 50 ml with eluting solvent, and the absorption at 450 m$\mu$ is measured. Results based on absorption at 450 m$\mu$ may correlate better with biological activity than those based on absorption at other wavelengths which indicate higher carotene contents.
S. C. JOLLY

**3778. A method for the separation of the constituents of pyrethrum extract.** R. G. W. Spickett (Colonial Products Lab., S. Kensington, London). *Chem. & Ind.*, 1957, (18), 561.—A method for the separation of "pyrethrin I" and "pyrethrin II" from an extract of pyrethrum is described. A chromatographic column (2·5 cm $\times$ 60 cm) is packed with a mixture of silica gel and plaster of Paris (4:1) (140 g); purified pyrethrum extract (0·87 g) in 5 ml of solvent (15% ethyl acetate in *n*-hexane) is run on the top of the column and developed with the same solvent mixture. "Pyrethrin I" is eluted in the 150 to 250-ml fraction and "pyrethrin II" in the 425 to 625-ml fraction. Quant. recoveries are quoted.
H. B. HEATH

**3779. Polarographic analysis of commercial hexachlorocyclohexane.** V. Šedivec (Inst. Hyg. and Ind. Diseases, Prague, Czechoslovakia). *Chem. Průmysl*, 1956, **6** (10), 401–404.—The height of the polarographic wave at $-0\cdot 7$ V is proportional to the content of heptachloro*cyclo*hexanes, that at $-1\cdot 7$ V corresponds to the sum of the heptachloro*cyclo*hexanes and the *p*-isomer of hexachloro*cyclo*hexane. From these data the amounts of both components can be easily determined. *Procedure*—Dissolve the sample (1 g) in acetone (25 ml), and to a 2-ml aliquot add acetone (3 ml), 0·5 M KCl (2 ml), gelatin soln. (0·1%) (1 ml) and water (to 10 ml). Register the polarographic wave.
J. BÖSWART

**3780. Quantitative determination of the gamma isomer of hexachlorocyclohexane by an infra-red absorptiometric method.** B. Kontnik and T. Kraczkiewicz (Dept. of Technical Physics, Inst. of General Chemistry, Warsaw). *Chem. Anal., Warsaw*, 1956,

**1** (4), 311–319.—The $\gamma$-isomer in the technical material obtained in the chlorination of benzene is determined by dissolving the sample in nitromethane and measuring the absorption of the soln. at 11·825 $\mu$. To correct for the interference due to the $\alpha$-isomer, its absorption value at 11·550 $\mu$, which equals its absorption at 11·825 $\mu$, is determined and subtracted from the value measured at 11·825 $\mu$. The relative error of this method, as compared with results obtained by the polarographic method, did not exceed 3%.    K. F. SPOREK

**3781. [Simultaneous] infra-red determination of dichlorodiphenyltrichloroethane [dicophane] and [gamma] benzene hexachloride [hexachlorocyclohexane] in insecticides.** W. B. Bunger and R. W. Richburg (Dept. of Chem., Alabama Polytech. Inst., Auburn, U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (2), 127–130.—A rapid routine method, based on infra-red absorption, is described for the simultaneous determination of dicophane (*pp′*-isomer) (**I**) and $\gamma$-hexachloro*cyclo*hexane (**II**) in cotton dust insecticides containing a high percentage of other constituents such as S, talc and clay. The sample (5 g) is extracted with $CS_2$ in a simple fat-extraction apparatus, and the absorption of the extract in the region from 8 to 15·4 $\mu$ is recorded. The absorption band of **I** at 9·8 $\mu$ and that of **II** at 14·53 $\mu$, both corrected for interference, are compared with standard curves. An average error of ±0·05% was obtained for single determinations.    S. C. JOLLY

**3782. Determination of tetramethylthiuram disulphide and dimethyldithiocarbamate spray residues on apples.** H. E. Barr, P. J. Clark and H. Jacks (Dominion Lab., Wellington, N.Z.). *N.Z. J. Sci. Tech.*, B, 1957, **38** (5), 425–432.—A micro-method capable of detecting 0·1 p.p.m. of tetramethylthiuram disulphide (thiram) and the dimethyldithiocarbamate compound of Fe and Zn in spray residues on apple skins is described. The skins are extracted with $CHCl_3$, the extracts are refluxed with 2 N $H_2SO_4$, followed by steam-distillation of dimethylamine with excess of alkali, and formation of yellow cupric dimethyldithiocarbamate by the method of Dowden (*cf. Biochem. J.*, 1938, **32**, 455). The intensity of colour is determined at 434 m$\mu$.    H. B. HEATH

See also Abstracts 3752, 3760.

# 5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

## General

**3783. Small dry box for sampling.** E. C. Fiebig, E. L. Spencer and R. N. McCoy (Shell Dev. Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 861.—A common glass funnel, 150 mm in diam., which has been adapted for dry sampling, is described and illustrated. The sample vial holder is shown. Operations are conducted through an aperture 16 mm × 50 mm cut in the side of the funnel, through which inert gas at positive pressure escapes continually.    E. G. CUMMINS

**3784. Automatic constant-level device for liquid nitrogen.** D. E. Henshaw (Univ. of Western Australia, Nedlands). *J. Sci. Instrum.*, 1957, **34** (5), 207–208.—A float in the Dewar vessel operates a mercury switch, which in turn operates a solenoid closing a valve in the gas vent of the supply vessel and operating a heater in this vessel. The increase in gas pressure forces over liquid nitrogen through a siphon.    G. SKIRROW

**3785. Improvements in or relating to apparatus for measuring, indicating or recording the presence or concentration of a combustible gas in an atmosphere.** Coal Industry (Patents) Ltd. [Inventors: W. H. Walton and F. W. Pritchard]. Brit. Pat. 778,774; date appl. 1.12.54.—An apparatus is described for measuring the change in the heat produced by a flame resulting from a change in the combustible gas or vapour concn. in the atmosphere. Both the rate of supply of fuel to the flame and the heat production are measured and controlled, and use is made of both these factors in obtaining a measurement of the gas or vapour concn. in the atmosphere.    N. E.

**3786. Apparatus for the continuous automatic measurement of the quantity of a certain gas in a gas mixture.** CKD-Dukla N.P. [Inventors: N. Celiscev and G. Held]. Brit. Pat. 779,824; date appl. 6.8.54.—The invention relates to apparatus for measuring, for instance, the percentage of $CO_2$ in combustion gases by the use of two flow-meters, one in front of, and the other behind, a reaction device in which the gas is absorbed; the quantity of the gas component is calculated from the difference in the readings of the two flow-meters.    N. E.

**3787. Improvements in or relating to testing air or other gases to determine the methane content thereof.** O. H. Drager. Brit. Pat. 773,534; date appl. 10.3.54. Germany, date appl. 13.3.53.—Apparatus for testing air or other gases to determine their methane content comprises an indication tube containing successive layers of an ozone-forming reagent (*e.g.*, silica gel containing $H_2SO_4$ and a permanganate) and a formaldehyde-indicating reagent (chromotropic acid). The indication tube has a polyethylene cap which is broken when the apparatus is to be used.    J. M. JACOBS

**3788. Warburg manometer flask for increased sensitivity in ethylene determinations.** H. K. Pratt and C. W. Greiner (Univ. Calif., Davis, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 862.—An improved flask is described for the manometric determination of ethylene by the method of Young *et al.* (*Anal. Chem.*, 1952, **24**, 551). A minimum of 44 $\mu$l of ethylene can be determined as opposed to the minimum of 120 $\mu$l possible with the standard vessels.    G. P. COOK

**3789. Improved automatic fraction collector.** G. F. H. Box and R. B. Bradbury (Div. of Ind. Chem., C.S.I.R.O., Melbourne). *J. Sci. Instrum.*, 1957, **34** (5), 183–184.—The collector described is designed to deliver constant volumes. Liquid flows into the measuring chamber, the lower end of which is sealed by a ball-valve. When a predetermined quantity of liquid has been delivered, a beam of light incident on a phototransistor is interrupted and the ball-valve is automatically opened, while delivery to the chamber is at the same time stopped.    G. SKIRROW

**3790. Filter flasks.** British Standards Instn. (2 Park St., London). B.S. 1739:1957, 10 pp.—This revised British Standard specifies a range of filter flasks suitable for general laboratory use.
N. E.

**3791. Rapid method combustion tubes (Belcher and Ingram type).** British Standards Instn. (2 Park St., London). B.S. 1428:Part A5:1957, 9 pp.—Two combustion tubes are specified for the micro-determination of elements by the Belcher and Ingram method (*Anal. Chim. Acta*, 1950, **4**, 118); type 1 for carbon and hydrogen and type 2 for halogens and sulphur.
N. E.

**3792. Use of zirconium crucibles for peroxide fusions.** H. E. Blake, jun., and W. F. Holbrook (Northwest Electrodevelopment Exp. Sta., Bur. of Mines, Albany, Oregon). *Chemist Analyst*, 1957, **46** (2), 42.—Crucibles pressed from 0·0035-in. zirconium sheet of high purity (0·1% of hafnium) show almost imperceptible erosion after 20 to 50 fusions. Comparative graphs of the average losses after peroxide fusions in crucibles of nickel, iron, zirconium and porcelain show the superiority of zirconium. The losses sustained by five crucibles after 18 to 54 fusions are tabulated.
W. J. WRIGHT

**3793. Improvements in or relating to viscosity meters.** K. T. Kalle. Brit. Pat. 778,519; date appl. 16.1.56. Sweden, date appl. 25.2.55.—The invention relates to a viscometer intended for continuously indicating the viscosity or concn. of a flowing liquid; it comprises a rotary member rotatable round a vertical axis, tapered at its lower free end and partly submerged in the flowing liquid. The resistance against its rotation caused by the varying viscosity of the liquid is measured.
N. E.

## Optical

**3794. Monochromatic diffraction-absorption technique for direct quantitative X-ray analysis.** D. H. Lennox (Dept. of National Health and Welfare, Ottawa, Canada). *Anal. Chem.*, 1957, **29** (5), 766–770.—Direct quant. analysis of α-quartz, with a Norelco diffractometer and either a monochromatic beam, diffracted by a crystal monochromator, or the polychromatic beam emitted by a copper-target X-ray tube, is described. The disadvantages of the latter method are discussed, and both techniques are compared with the internal-standard method.
E. G. CUMMINS

**3795. Improvements in or relating to spectrometers.** G. Touvet. Brit. Pat. 774,473; date appl. 12.1.51.—The optical radiation used is modulated at radio frequency. A part of the radiation corresponding to one or more wavelengths is selected, and the modulations of these parts are converted into variations in the intensity of an electric current or voltage. The amplitude of these variations affords a measure of the intensity of the selected part of the radiation.
J. M. JACOBS

**3796. Improvements in and relating to spectrometers.** C. A. Parsons and Co. Ltd. [Inventor: A. E. Martin]. Brit. Pat. 779,199; date appl. 1.6.54. —The invention relates particularly to i.r. spectrophotometers in which a plane diffraction grating is used for the production of spectra. The grating can be rotated in such a way that a linear relation is obtained between wavelength and the displacement of a screw, which is so calibrated as to indicate the wavelength directly in microns.
N. E.

**3797. Simple logarithmic ratio-recording spectrophotometer attachment.** P. Hariharan and M. S. Bhalla (Nat. Phys. Lab. of India, Delhi). *J. Opt. Soc. Amer.*, 1957, **47** (5), 378–381.—The apparatus described, used in conjunction with a recording potentiometer, enables a monochromator to be converted into a recording spectrophotometer. A double-beam system is used with a single photomultiplier tube, thus avoiding the necessity of matching the spectral sensitivities of two tubes. The electrical circuits are designed to give direct readings of the logarithm of the spectral transmission, optical densities as high as 3 being measured with an error not exceeding 0·02. The spectral range of the instrument is governed by the characteristics of the photomultiplier used; by a suitable choice of photomultiplier type, any region of the visible or u.v. spectrum may be selected.
B. S. COOPER

**3798. Spectrographic procedure for analysis of powdered samples of unknown origin.** H. E. Biber and S. Levy (U.S. Steel Appl. Res. Lab., Monroeville, Pa., U.S.A.). *J. Opt. Soc. Amer.*, 1957, **47** (5), 381–385.—Equal weights (usually 5 to 30 mg) of the sample and of pure graphite powder containing 1% of $GeO_2$ are mixed together. A portion of this prepared sample is packed into a crater, 1 mm diam., 1 mm deep, in a 3/16-in. graphite rod. With this sample electrode positive, the mixture is burnt to completion in a d.c. arc of 4 amp. in 2 min. Germanium is used as the internal standard and calibration curves are prepared either by using synthetic mixtures or samples of established composition. The useful concn. ranges cover both minor and major constituents, the average accuracy for duplicate determinations being ±15%. Suitable line pairs are given for—Al, Sb, As, Bi, B, Ca, Cr, Cu, Fe, Pb, Mg, Mn, Ni, P, Si, Ag, Na, Sn, Ti and Zn. The method also permits the determination of the elements in a very small sample ($\simeq 1$ mg).
B. S. COOPER

**3799. A recording spectrophotometric titrimeter.** R. A. Chalmers and C. A. Walley (Dept. of Geology, Durham Colleges, Durham). *Analyst*, 1957, **82**, 329–336.—An apparatus is described and illustrated for the determination of titrimetric end-points by the automatic recording of the rate of change of the extinction of the soln. being titrated. It is designed for the titration of microgram amounts, either in very dil. soln. or in small amounts of more conc. soln. Examples are given of the titration of HCl with NaOH (indicator, bromocresol purple), $CaCl_2$ with EDTA (indicator, murexide), and $MgCl_2$ with EDTA (indicator, Solochrome black WDFA). The accuracy is such that, in general, it is possible to titrate with not more than 0·5 ml of titrant with a reproducibility to 0·0015 ml.
A. O. JONES

**3800. Method of obtaining derivative spectra.** J. P. Pemsler (Goodyear Atomic Corp., Portsmouth, Ohio, U.S.A.). *Rev. Sci. Instrum.*, 1957, **28** (4), 274–275.—Overlapping spectral bands may be detected by using the first derivative with respect to wavelength of the transmission curves. The Perkin - Elmer model 13 can be adapted to record such spectra directly.
G. SKIRROW

**3801. Device for monitoring absorbance of column eluates in a spectrophotometer.** J. B. Stark, R. Teranishi and G. F. Bailey (U.S. Dept. of Agric., Albany, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 861–862.—Details are given of a cell assembly suitable for continuous monitoring of a chromatographic column eluate.    E. G. CUMMINS

**3802. Infra-red grating spectrophotometer.** J. U. White, N. L. Alpert, A. G. Debell and R. M. Chapman (White Development Corp., Stamford, Conn., U.S.A.). *J. Opt. Soc. Amer.*, 1957, **47** (5), 358–367.—A spectrophotometer, based on the double-beam optical null principle and incorporating a grating and fore prism double monochromator, is described. The instrument is capable of very high resolution, the grating being usable in any order with either single or double pass of the grating monochromator. The spectra are recorded directly in terms of transmission *vs.* wavelength; the performance is illustrated by the spectra of water vapour and of $N_2O$.    B. S. COOPER

**3803. Apparatus for analysing liquids which contain a crystalloid substance and a non-crystalloid substance [particularly body fluids for diagnostically significant factors].** Technicon International Ltd. [Inventor: L. T. Skeggs]. Brit. Pat. 774,711; date appl. 13.9.54.—In the determination, *e.g.*, of glucose, the sample is pumped through a dialyser, the dialysate is mixed with a solution of $K_3Fe(CN)_6$, the mixture is examined photometrically and the result is recorded automatically. Samples are treated in a continuous segmented stream in which successive samples are separated by air segments.    J. M. JACOBS

**3804. The production of slides of emulsions.** A. W. Middleton (33 Devereux Drive, Watford, England). *Analyst*, 1957, **82**, 289.—A method is described for preparing slides of emulsions without altering the size or shape of the globules of the disperse phase or splitting the emulsion. It consists essentially in tying round the slide two wires of known thickness separated by a distance less than the diameter of the cover glass. A drop or two of the emulsion is placed between the wires and the cover glass is then gently pressed upon the two wires. Slides of any thickness can thus be made and the min. thickness that will give a true picture of the emulsion can be determined. A list of the diameters of suitable wires is given with their corresponding British Imperial Gauge numbers.    A. O. JONES

### Thermal

**3805. Thermistor probe.** W. Belfield and R. W Johnson (Univ. Coll. of the Gold Coast, Accra). *J. Sci. Instrum.*, 1957, **34** (5), 209.—A fine probe suitable for measuring temp. of 20° to 30° with an accuracy of 0·1° was constructed by mounting a thermistor in the tip of a hypodermic needle 0·8 mm in diam.). Details of the method of mounting are given.    G. SKIRROW

**3806. Automatic recording thermobalance.** C. Groot and V. H. Troutner (Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 835–839.—The design of a thermobalance used to study the composition of hydrated $Al_2O_3$ samples by measuring weight loss is described. The modification of an analytical balance is illustrated and the calibration and characteristics of the thermobalance are discussed.    E. G. CUMMINS

**3807. Adiabatic calorimeter for determination of cryoscopic data.** S. V. R. Mastrangelo (Allied Chemical and Dye Corp., Glenolden, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 841–845.—A precision adiabatic calorimeter for determining cryoscopic data of compounds melting in the range −175° to +140° is described. The m.p., heat of fusion and cryoscopic constants of phenol, 3:5-xylenol, *p*-α-cumylphenol, quinoline, 2:4:6-collidine, ε-caprolactam, maleic anhydride, di-α-cumyl peroxide and naphthalene are presented. Freezing-point data for systems comprising these compounds and probable impurities enable a sound routine evaluation of purity to be made when the impurities follow Raoult's law.    E. G. CUMMINS

**3808. Thermodynamic analysis. IX. An air or gas calorimeter.** W. Oelsen. *Arch. Eisenhüttenw.*, 1957, **28** (1), 1–6.—A calorimeter was developed from which enthalpy curves are obtained by continuous measurement, by thermocouples, of the temp. change of a gas stream passing over the sample. The inlet gas is at room temp. The apparatus is always ready for use without preparation; the procedure is about 100 times as rapid and yields results at least as reliable as those by classical methods. Good results were obtained for the heat of fusion of metals, including Hg, quantitative analysis of alloys, transition temp. of a sample of steel and the sp. heat of gases.    G. BURGER

**3809. Modified Hershberg melting-point apparatus.** H. E. Drechsel (Smith, Kline and French Lab., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (5), 859–860.—A U-tube device incorporating a thermometer calibrated *in situ* is illustrated. Acceleration of heating is constant, and depends on the voltage input supplied through a variable transformer; a setting of 10 to 55-V input gives a temperature range of 45° to 320° under normal conditions.    E. G. CUMMINS

**3810. Vacuum-fusion gas-analysis apparatus for use in the quality control of ferrous materials.** K. Speight and G. M. Gill [Metallurgy (General) Division, B.I.S.R.A., Sheffield]. *Metallurgia, Manchr*, 1957, **55**, 155–158.—The mixture of gases (CO, H, N) evolved by melting a specimen of ferrous metal passes to a combined Toepler pump (operated with an air compressor) and McLeod gauge. From the calibrated bulbs, the gas circulates to a CuO catalyst (at 600°) where CO is oxidised to $CO_2$ and H to $H_2O$. The water is removed in a trap containing $P_2O_5$ before the gases enter a freezing trap. By measuring the gas pressure before and after the oxidation and water-absorption stage, the content of H of the metal is obtained. The contents of O and N are determined by freezing out the $CO_2$ (immersing the trap in liq. O), pumping off residual N, and allowing the $CO_2$ to expand to room temp. and measuring its pressure.    S.C.I. ABSTR.

### Electrical

**3811. Quantitative microscopic ionophoresis and chromatography.** B. M. Turner (Turner Lighter Spares and Components, Ltd., Hendon Central, London). *Nature*, 1957, **179**, 964–965.—An apparatus and procedure for microchromatography and

electrophoresis are described, in which liquid films ($0 \cdot 01$ to $10 \ \mu$) on a clean polished microscope slide are used. Drops ($10^{-4}$ to $10^{-6}$ litre) of the mixture of cations are delivered to the under-side of the slide and the vol. is determined from the circular reflection interference fringes produced by monochromatic green light. A suitable supporting liquid (HCl, Teepol, trichloroacetic acid) is then deposited on the film and, after equilibrium is reached, a d.c. at 30 to 70 V per cm is applied for 20 to 180 sec. through platinum-wire electrodes in contact with the slide. Ammonia vapour is then admitted, the film is dried and the resolved cationic zones are revealed by the "breath-figure" test (*cf.* Sonders *et al.*, *J. Appl. Phys.*, 1950, **21**, 338) and their areas are measured. The analysis is made in a glass humidity-cell on a platform attached to a microscope vernier stage; a 0·5 to 1-in. objective and a 30 to 375 magnification are used. The method, which is claimed to have a sensitivity $\simeq 10^{6}$ times as great as that of paper-strip methods, should facilitate electrophoresis at cellular or sub-cellular levels.                       W. J. BAKER

**3812. Paper electrophoresis in a damp chamber. V. Continuous electrophoresis involving obstruction of the electrodes with membranes.** Z. Pučar (Inst. "Ruder Bošković," Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1957, **29** (1), 1–5.—The adhesion of Cellophane strips to the electrodes in paper electrophoresis in a damp chamber causes fundamental changes in the horizontal and vertical components of the electrode channels. These are discussed. The closing of these channels is of significant importance in a continuous process in that it prevents loss of buffer soln. or electrolyte.
W. J. WRIGHT

**3813. Oscillographic polarography.** J. Heyrovský (Polarograph. Inst., Czechoslovak Acad., Prague). *Öst. ChemZtg*, 1957, **58** (7–8), 94–99.—A description is given of the principles, apparatus and new applications of the method.     H. F. W. KIRKPATRICK

**3814. The aluminium indicator-electrode in acid-base potentiometric titrations.** E. Scarano (Inst. Anal. Chem., The Univ., Rome, Italy). *Anal. Chim. Acta*, 1957, **16** (4), 365–369 (in English).—Reproducible and accurate acid-base potentiometric titrations can be made with aluminium indicator-electrodes provided that the surface of the electrodes is pretreated, preferably by immersing the degreased electrode in boiling $Na_2HPO_4$ soln. for a few min. The range of potential during titration, *e.g.*, of K H phthalate with 0·1 or 0·01 $N$ NaOH, is $\simeq 3 \cdot 6$ times as great as for the antimony electrode, but the titration takes longer than with other metal electrodes.     W. J. BAKER

**3815. High-frequency titration with the Huth-Kühn circuit.** A. J. Arvia and P. H. Brodersen (Univ. Nac. Inst. de Fisica, La Plata, Argentine). *Z. physik. Chem.*, 1956, **6**, 381–386.—The regenerative valve oscillator with tuned grid and anode circuits often used for conductimetric titration (Huth-Kühn circuit) exhibits a region of instability close to the sensitivity max. The behaviour of the circuit was examined theoretically and experimentally. It was shown that the unstable region can be eliminated by increasing the internal resistance. This can be done by reducing the heating voltage or, better, by taking the anode and grid connections to tappings on the anode and grid coils. Circuit diagrams, curves showing the behaviour of the circuit, and plots of two titrations are presented.                       G. BURGER

**3816. Integrated-current source for automatic coulometric titrations.** L. E. Smythe (Anal. Chem. Group, A.E.R.E., Harwell, England). *Analyst*, 1957, **82**, 228–233.—The simple unit described for automatic coulometric titration can be used with a commercial automatic pH titrimeter and with wide variations in mains voltage. Provision is made for "fast" titration to near the end-point and a "slow" titration as the end-point is approached. The application of the equipment to the determination of Cr in $K_2Cr_2O_7$ soln. is described, and the quoted results show satisfactory accuracy. Equal accuracy was found with the determination of Cr in steel and of Ce in the presence of U and La. A. O. JONES

**3817. Improved instrument for high-frequency conductimetric titration.** F. Kupka and W. H. Slabaugh (Oregon State Coll., Corvallis, U.S.A.). *Anal. Chem.*, 1957, **29** (5), 845–848.—End-point detection in titrations involving colloidal electrolytes has been investigated with the instrument described, which is a modification of that of Blake (*cf.* "Conductimetric Analysis at Radio-frequency," Chemical Publishing Co., New York, 1952, p. 34). Linear response is obtained over the range of conductance of $4 \times 10^{-4}$ to $6 \cdot 5 \times 10^{-2} \ N$ KCl when fixed frequencies with an amplitude-stabilised oscillator and capillary-restricted conductance cells are used. Response curves and circuit diagrams are given. Unambiguous results are reported in the non-linear low-conductance ($3 \times 10^{-5} \ M$ KCl) and high-conductance (1 $M$ KCl) regions.
E. G. CUMMINS

**3818. Precision paramagnetic resonance spectrometer.** K. D. Bowers, R. A. Kamper and R. B. D. Knight (Clarendon Lab., Univ., Oxford). *J. Sci. Instrum.*, 1957, **34** (2), 49–53.—The instrument is designed for the accurate comparison in the same magnetic field of the frequency of electronic magnetic resonance absorption with the frequency of proton resonance absorption. The field can be stabilised at any value between 1 and 11 kG. The microwave frequency is stabilised in the region 9000 to 10,000 Mc/s and the paramagnetic resonance absorption is detected at this frequency using 115 kc/s magnetic field modulation. The precision is about 3 p.p.m.     G. SKIRROW

**3819. New method for the preparation of thin films of radioactive material.** D. J. Carswell and J. Milsted (A.E.R.E., Harwell, England). *J. Nuclear Energy*, 1957, **4** (1), 51–54.—An acetone soln. of an α-active compound is expelled from a very fine capillary mounted vertically above a metal disc by applying a voltage of 3 to 10 kV between the soln. (positive) and the disc, on which the nuclide is deposited. The resolution of the α-particle energies is equal to that obtained by using sources prepared by vacuum-evaporation methods. The recovery of activity is approx. 80%.     G. J. HUNTER

ERRATA.—June (1957) issue, abstract 1765, line 4.  *For* "1956, **1** (1), 15–20" *read* "1956, **7** (1), 15–20."
August (1957) issue, abstract 2579, line 1.  *For* "Photometric" *read* "Potentiometric."
August (1957) issue, abstract 2620, line 4.  *For* "D. Mappin" *read* "D. Mapper."

# ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

| | | |
|---|---|---|
| alternating current | . | a.c. |
| ampere | . | amp. |
| Ångström unit | . | A |
| anhydrous | . | anhyd. |
| approximate, -ly | . | approx. |
| aqueous | . | aq. |
| atmospher-e, -ic. | . | atm. |
| boiling-point | . | b.p. |
| British thermal unit | . | B.Th.U. |
| calorie (large) | . | kg-cal. |
| calorie (small) | . | g-cal. |
| centimetre | . | cm |
| coefficient | . | coeff. |
| concentrated | . | conc. |
| concentration | . | concn. |
| constant | . | const. |
| corrected | . | (corr.) |
| crystalline. | . | }cryst. |
| crystallised | . | |
| cubic | . | cu. |
| current density | . | c.d. |
| cycles per second | . | c/s |
| density | . | $\rho$ |
| density, relative | . | $d$ or wt. per ml |
| dilute | . | dil. |
| direct current | . | d.c. |
| distilled | . | dist. |
| ethylenediaminetetra-acetic acid | . | EDTA |
| electromotive force | . | e.m.f. |
| equivalent | . | equiv. |
| gram | . | g |
| gram-molecule | . | mole |
| half-wave potential | . | $E_{\frac{1}{2}}$ |
| hour | . | hr. |
| hydrogen ion exponent | . | pH |
| infra-red | . | i.r. |
| insoluble | . | insol. |
| international unit | . | i.u. |
| kilogram | . | kg |
| kilovolt | . | kV |
| kilowatt | . | kW |
| liquid | . | liq. |
| maxim-um, -a | . | max. |
| melting-point | . | m.p. |
| microgram | . | $\mu$g (not $\gamma$) |
| microlitre | . | $\mu$l |
| micromole | . | $\mu$mole |
| micron | . | $\mu$ |
| milliampere | . | mA |

| | | |
|---|---|---|
| milli-equivalent | . | milli-equiv. |
| milligram | . | mg |
| millilitre | . | ml |
| millimetre. | . | mm |
| millimicrogram | . | m$\mu$g |
| millimolar. | . | m$M$ |
| millivolt | . | mV |
| minute (time) | . | min. |
| molar (concentration) | . | $M$ |
| molecul-e, -ar | . | mol |
| normal (concentration) | . | $N$ |
| optical rotation | . | $\alpha_{\lambda}^{t}$ |
| ounce | . | oz |
| parts per million | . | p.p.m. |
| per cent. | . | % |
| per cent. (vol. in vol.) | . | %(v/v) |
| per cent. (wt. in vol.) | . | %(w/v) |
| per cent. (wt. in wt.) | . | %(w/w) |
| potential difference | . | p.d. |
| precipitate (as a noun) | . | ppt. |
| precipitated | . | pptd. |
| precipitating | . | pptg. |
| precipitation | . | pptn. |
| preparation | . | prep. |
| qualitative, -ly | . | qual. |
| quantitative, -ly | . | quant. |
| recrystallised | . | recryst. |
| refractive index | . | $n_{\lambda}^{t}$ |
| relative band speed | . | $R_{F}$ |
| relative humidity | . | r.h. |
| revolutions per minute | . | r.p.m. |
| saponification value | . | sap. val. |
| saturated calomel electrode | . | S.C.E. |
| second (time) | . | sec. |
| soluble | . | sol. |
| solution | . | soln. |
| specific gravity | . | sp. gr. |
| specific rotation | . | $[\alpha]_{\lambda}^{t}$ |
| square centimetre | . | sq. cm |
| standard temp. and pressure | . | s.t.p. |
| temperature | . | temp. |
| ultra-violet | . | u.v. |
| vapour density | . | v.d. |
| vapour pressure | . | v.p |
| volt | . | V |
| volume | . | vol. |
| watt | . | W |
| wavelength | . | $\lambda$ |
| weight | . | wt. |

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

| | | | |
|---|---|---|---|
| greater than | . | . | $>$ |
| not greater than | . | . | $\not>$ |
| is proportional to | . | . | $\propto$ |

| | | | |
|---|---|---|---|
| less than | . | . | $<$ |
| not less than | . | . | $\not<$ |
| of the order of, approximately | | | $\simeq$ |

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, $Fe^{II}$, $Mo^{V}$. Substances in the ionic state are represented by $Na^{+}$, $Fe^{2+}$, $Fe^{3+}$, etc., for cations and by $Cl^{-}$, $SO_4^{2-}$, $PO_4^{3-}$, etc., for anions.

# ANALYTICAL ABSTRACTS

## A PUBLICATION OF
## THE SOCIETY FOR ANALYTICAL CHEMISTRY

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